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PAKISTAN JOURNAL OF SCIENTIFIC AND INDUSTRIAL RESEARCH

Vol. 4, No. 1 January 1961

CONTENTS

Constituents of Rauwolfia Vomitoria from East Pakistan.....Salimuzzaman Siddiqui and M. Manzur-i-Khuda 1

Roof Slabs in Low-Cost Houses.....S. Tehzibul Hasan 4

Utilisation of 'Balcrete' Foaming Agent. Part I.—Production of Cellular Concrete (with Neat Cement).....Mohamad Aslam, S. Tehzibul Hasan, Mubarak Ahmad and M. Jehangir 9

A Gravity Survey of Quetta and Mastung Valleys.....Irshad Mufti and F. A. Siddiqui 15

Preliminary Survey and Chemical Investigations on Iron Ores of Khyber Agency..M. Nabi Bukhsh and Fazal Muhammad 21

A Study of Multani Mitti.....Ainul Hasan Khan 25

An Investigation of the Influence of Humidity and Water Content on the Electrical Properties of Cottonseed Cake.....A. K. Bhuiya and Mazhar M. Qurashi 29

Chemical Studies in Relation to the Biosynthesis of Some Natural Benzofurans and Related Compounds. Part II......B. I. Nurunnabi 34

Attempted Synthesis of Evodone.....B. I. Nurunnabi 37

Standardization of Some Local Aromatic Herbs. Part I......M. A. Wahid and M. Ikram 40

Special Article:

Summarized Report on the Proceedings of the 4th Congress of the Pan Indian Ocean Science Association and the Recommendations of its Council.....M. Qudrat-i-Khuda, A. H. Chotani and Mazhar M. Qurashi 42

Short Communications:

OBSERVATIONS ON THE VOLTAGE DEPENDENCE OF THE INSULATION RESISTANCE OF SOME INSULATING MATERIALS.....S. Nurul Ahmad and Mazhar M. Qurashi 49

A NOTE ON THE OCCURRENCE OF PULLET DISEASE: AVIAN MONOCYTOSIS IN EAST PAKISTAN.....M. Wazed Ali 51

Book Notices..... 52

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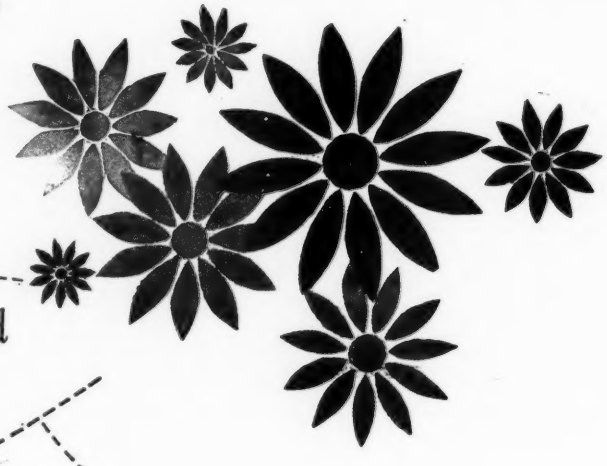

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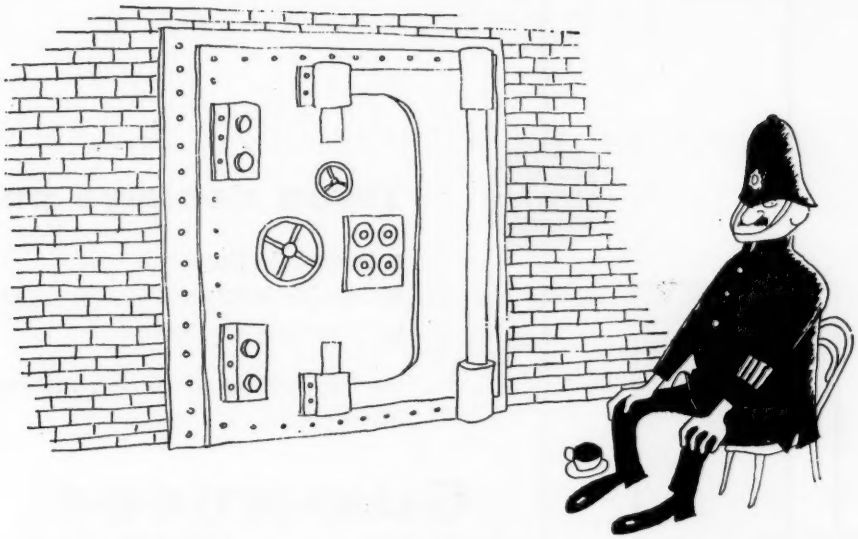

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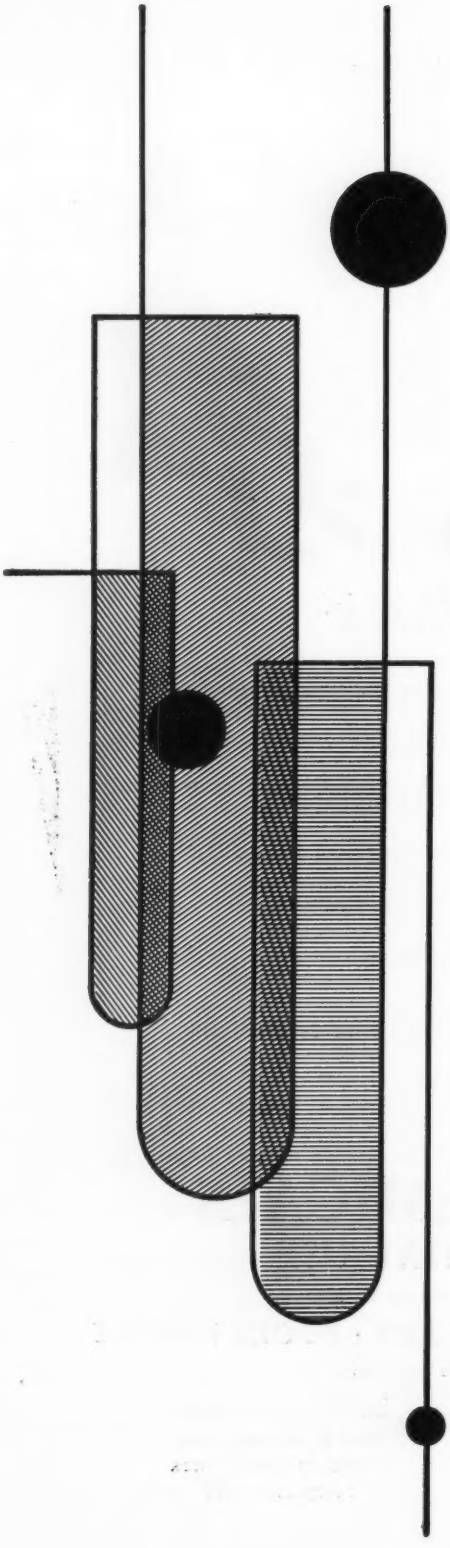

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CONSTITUENTS OF RAUWOLFIA VOMITORIA FROM EAST PAKISTAN

SALIMUZZAMAN SIDDIQUI AND M. MANZUR-I-KHUDA

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(Received December 22, 1960)

R. vomitoria is native to French Guinea and is histologically similar to the American species *Rauwolfia heterophylla Roem. and Schult.*

Paris[1] reported the isolation of the ajmaline series of bases,[2] ajmaline, *iso*-ajmaline, small quantities of ajmalinine and ajmalicine along with traces of a yellow base, from the roots of the plant grown in French West Africa. From the roots of the same plant Schlittler and co-workers[3] isolated alstonine.[4,5,6] Poisson and co-workers[7] reported the isolation of reserpine[7] and two new alkaloids namely raumitorine and seredine.[8] In 1955, Haack and co-workers islolated rauvomitine[9] and Kidd reported the isolation of rescinnamine[10] from the same plant. *Iso*-reserpiline and serpagine were isolated from it in 1955.[11]

In view of the interesting results obtained with the alkaloidal complex serpajmaline as a hypotensive agent,[12,13] the present work was undertaken for isolating the active principles of the plant in the form of their naturally occurring complexes and investigating their chemical constituents. The extracts of the fresh undried drug material were worked up as outlined in the experimental to give three alkaloidal complexes, of which the water soluble complex I corresponded to serpajmaline isolated from *Rauwolfia serpentina*, and had marked hypotensive action on dogs. The action of the drug was prolonged and the material from root bark was found to be more potent than that from the whole root. Like serpajmaline it also had antifibrillant action on the isolated heart of rabbit. This complex was found to contain alstonine and ajmaline as the major alkaloidal constituents, along with traces of serpentinin and an undefined amphoteric base. With modified working conditions it also gave a hydroiodide which showed green fluorescense.

Complex II was isolated as a pale yellow powder, insoluble in pertoleum ether, benzene, ether and water. It gave ajmaline and reserpine together with a crystalline material which could be identified as serposterol.

Complex III was obtained as a petroleum ether soluble fraction which gave ajmaline, reserpine and serposterol on chromatography over alumina. Four sugars, maltose, sucrose, glucose and mannose were identified from the aqueous ballast on paper chromatography.

Experimental

The material for this work was obtained tarough the courtsey of Dr. S. Hedayetullah, from an experimental plantation in Dacca.

Isolation of the Alkaloidal Complexes.—Freshly harvested roots (moisture content 55%; total alkaloids 1.7% on moisture free basis) were chopped into approximately 1 cm. long pieces (1/2 kg.) and repeatedly extracted as such with ethanol (95%, 2 ½ l. × 1; 80%, 2 ½ l. × 1; 70%, 2 ½ l. × 2). The combined extracts were concentrated to semisolid residue (18 g.) below 40°C. in vacuo.

Fresh root bark (1 kg., moisture content 70%; total alkaloids 1.6 % on moisture free basis) was also similarly extracted with ethanol (2 l. × 5) to give a dark green semisolid residue (25.0 g.) after removing the solvent. This (25.0 g.) was triturated with water and ethyl acetate. The

a queous layer was shaken out wtih water and e xtracted with amyl alcohol (3×). The combined amly alcohol extracts were then repeatedly shaken out wit h water. The aqueous extract on removal of the solvent in vacuo gave a cream yellow powder designated as complex I (5 g.). The ethyl acetate layer was dried over Na_2SO_4 and freed of the solvent in vacuo and the semisolid residue exhaustively extracted with petroleum ether. The petroleum ether extracts gave on removal of the solvent a fatty residue designated as complex III. The petroleum ether insoluble residue formed a cream coloured powder designated as complex II.

Constituents of Complex I.—Five g. of this complex was dissolved in a small amount of water and the aqueous solution (pH 4–5) was treated with dilute ammonia (1%) to pH 7.5 and repeatedly extracted with ethyl acetate (7×). The ethyl acetate solution, dried over Na_2SO_4, gave on removal of the solvent a glassy mass which, after charcoaling in methanol, slowly crystallised from dilute methanol to give ajmaline, m.p. 152°C., twice crystallised from the same solvent it gave m.p. 158°C. (Found: C, 71.85; H, 7.95; O, 11.7. $C_{2c}H_{2c}O_2N_2.-\frac{1}{2}H_2O$ requires C, 71.5; H, 8.05; O, 11.9). Mixed m.p. with ajmaline undepressed (m.p. 158°C.) $[\alpha]^{22}_D$+ 125° (95% ethyl alcohol). Literature value $[\alpha]^{33}_D$ + 128°.

The aqueous layer from the above working was concentrated in vacuo and made acid with 10% acetic acid and treated with potassium iodide. The semi-crystalline precipitate thereby obtained was washed with a little water and crystallised from methanol to give alstonine hydroiodide, m.p. 282-4°C. decomp. (0.96 g.), twice crystallised from the same solvent it gave glistening colourless plates m.p. 286°C. decomp. (Found C, 52.95; H, 4.6; N, 5.6; O, 10.45; I, 26.8. $C_{21}H_{20}O_3N_2.-$HI requires C, 52.95; H, 4.45; N, 5.85; O, 10.1; I, 26.7). It had $[\alpha]^{27}_D$ + 162° in absolute ethanol. Literature m.p. of alstonine hydroiodide, 292°C. decomp. (corrected). The free base was obtained by adding dilute sodium carbonate solution to an aqueous solution of the hydroiodide. The base which is known to have a very unstable character had to be handled with great care in the process of isolation. It crystallised as light orangish yellow needles which decomposed above 150°C. without melting or sintering on slow heating. By decomposing the hydroiodide solution with dilute caustic soda no crystals could be obtained; adding it dropwise until the pH reached 8 resulted in deposition of violet needles. Alstonine decomposed slowly even when kept in a desiccator in vacuo (Found C, 69.45; H, 5.85; N, 8.0; $C_{21}H_{20}O_3N_2.\frac{1}{2}H_2O$ requires C, 68.85; H, 6.05; N, 7.651. Salts prepared from the free base failed to crystallise.

The aqueous filtrate from alstonine hydroiodide was made strongly alkaline with dilute caustic soda. A yellow precipitate thrown out was collected, washed with water and crystallised (charcoaled) from dilute ethanol to give serpentinine, m.p. 276°C. undepressed on admixture with an authentic specimen.

In a modified working based on liberation of the total base with alkali and separation of the bases through the hydroiodides, a methanol insoluble crystalline iodide was obtained which melted at 257°C. and could not be studied further due to insufficient quantity of the material.

Alstonine Hydrochloride.—Alstonine hydroiodide was triturated with freshly precipitated silver chloride (washed free from nitrate) and filtered warm. The residue was washed twice with hot methanol and warm water. The filtrate was evaporated in vacuo, the residue dissolved in methanol and charcoaled. The methanolic solution was diluted with ether when it deposited long prismatic needles, m.p. 282 – 4°C. decomp. $[\alpha]^{21}_D$ + 140.5° in distilled water. (Found C, 64.45; H, 5.4; N, 7.25; Cl, 8.9. $C_{21}H_{20}O_3-N_2.$ HCl. $\frac{1}{2}H_2O$. requires C, 64.05; H, 5.65; N, 7.15; Cl, 9.00). Literature m.p. 286°C. decompr. (corrected). Literature value $[\alpha]^{22}_D$ + 139.9° in water.

Complex II.—The cream yellow product was triturated with 2% caustic soda solution and extracted with ethyl acetate. The sticky residue was then triturated with 5% acetic acid solution and extracted with ethyl acetate. This alternate treatment with alkali and then with acid was repeated till all the material distributed itself between ethyl acetate and the aqueous layer which was kept alkaline. The bulked ethyl acetate layer was dried over Na_2SO_4 and freed of the slovent in vacuo. It was transferred with benzene to a column of alumina and chromatographed with the same solvent followed by mixtures of benzene, ether, chloroform and methanol. The concentrated benzene and chloroform eluates readily crystallised in rhombs, which melted at 273°C. and showed no depression in m.p. when mixed with an authentic specimen of reserpine. The 25% benzene and chlor. form eluate also crystallised from dilute methanol to give ajmaline, m.p. 158°C. (mixed m.p. with an authentic specimen showed no depression). The rest of the chromatographic fractions did not crystallise.

Complex III.—The semisolid complex obtained from the root material was transferred on to a

column of alumina with petroleum ether (b.p. 60-80°C.) and chromatographed with the same solvent followed by mixtures of benzene, ether, chloroform and methanol. The 1:1 ether/benzene eluate crystallised to give a material of m.p. 152°C., mixed m.p. with serposterol (m.p. 150-52°C.), 151-2°C.

The 1:2 chloroform/benzene eluate gave a substance of m.p. 245°C., recrystallised ex. ethanol m.p. 275°C. mixed m.p. with reserpine m.p. 275°C. (Found C, 65.95; H, 6.7; N, 4.75; O, 21.9. $C_{33}H_{40}O_9N_2$ requires C, 65.15; H, 6.65; N, 4.6; O, 23.65) $[\alpha]_D^{22}+116°$, Literaure value $[\alpha]_D^{23}$ $-117°$; the end fractions collected with the same solvent as eluate gave crystals m.p. 158°C. (mixed m.p. with ajmaline 158°C.).

Paper Chromatography of Aqueous Ballast.—A mixture of butanol (10 ml.), glacial acetic acid (10 ml.) and water was shaken thoroughly and the organic layer allowed to separate. Spots of the aqueous ballast and different sugars (mannose, glucose, gulose, sucrose, rhamnose, fructose and maltose) were chromatographed on a filter paper sheet for 3 days. It was allowed to dry and then sprayed with 1% silver nitrate solution, allowed to dry again and sprayed with 1% caustic soda solution. The resulting four brown spots of different sugars from both the root and root bark material were found to correspond to the spots of mannose, glucose, sucrose and maltose.

Summary

Complexes corresponding to serpajmaline, ajmalexine and resajmaline as isolated from *Rauwolfia serpentina* have now been obtained from *Rauwolfia vomitoria* grown in an experimental plantation in East Pakistan. The complex corresponding to serpajmaline was found to be a strong hypotensive agent in dogs and also had antifibrillant action on isolated heart of rabbit. The chemical constituents of these complexes have been examined and the one corresponding to 'serpajmaline' was found to contain alstonine as the major alkaloidal constituent, together with ajmaline and serpentinine. The other two complexes gave reserpine, ajmaline and serposterol. The extracts were found to contain maltose, sucrose, glucose and mannose as the sugar constituents.

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ROOF SLABS IN LOW-COST HOUSES

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(Received January 8, 1961)

This paper describes the design and testing of a roof in which the flexural strength of the concrete has been used to minimize the use of steel (from 170 lb. to 34 lb. for a room of 10′ × 11′ inside dimensions). This reduces the cost considerably without affecting the comfort in the house. The high test load indicates that such roof can also be used for more than one storey buildings.

Introduction

Roofs have been given much attention in the construction of low-cost houses, as these involve, to a greater extent, the use of mild steel, at present an imported material. Economy has been effected in the use of steel and cement, either by reducing the thickness of the roof slab through improving the quality of concrete (making it more dense), or by laying plain concrete tiles over precast reinforced concrete battens. Another approach, using prestressed concrete planks, has been approved for refugee houses, and a factory for the production of such precast units on a large scale is going to be set up in Karachi very soon.

The above modifications have been based simply on economy in construction, and the important factor of thermal comfort was totally negelcted. Thus, it was found that through reduction in the thickness of slab and by making the concrete more dense, the thermal conductivity of the roofing unit increased considerably. As a result, the houses were very warm in the summer and cool in the winter, and consequently they were uncomfortable for a major portion of the year. The use of an additional insulation layer on top of such roof slabs is not feasible due to the extra expense in the low-cost houses. An attempt has therefore been made by the author to design and test a type of roofing that can cut down the cost without affecting comfort in the house. The design of the roof, which has been discussed in the following, reduces the quantity of steel from approximately 170 lbs. for the construction of a single room of 10′ × 11′ inside dimensions to about 34 lbs. without any change in the quantity of concrete. In an earlier communication[1] on low-cost roofing for large-scale production of small houses, it has been mentioned that the precast units need a proper water-proofing layer. As the cost of this water-proofing layer is considerable, the roof has been designed as cast-in-situ to eliminate this, and it needs approximately the same amount of centering as in the conventional system. The constructional details are given in Fig. 1.

Design

In a reinforced concrete slab, the strength of cement concrete is generally neglected on the tension side and mild steel bars are provided to take the entire tensile stresses. In the case of this design, the flexural strength of the concrete has been utilized to reduce the amount of steel required on the tension side of a slab. The roof (10′ × 11′ inside) has been divided into three spans of plain concrete sections by providing two reinforced concrete beams projecting 6″ × 6″ below the ceiling level. As the beams have been designed on a rectangular section, these projections can be placed on the top of the roof if so desired, thus improving the appearance of the ceiling.

For calculating the amount of the stresses induced in the slab and beams, let M_A, M_B, M_C & M_D denote bending moments in foot pounds and R_A, R_B, R_C & R_D reactions in pounds at the supports A, B, C and D, respectively (Fig. 2); A and D are wall supports while B and C are beams, l_1 and l_2 being the effective spans of the slab in feet. W is the total weight (including superimposed load) in pounds per square foot area of the slab.

$$M_A = M_D = O \text{ and } M_B = M_C$$

$$2l_1 + l_2 = 10.5 \text{ ft.}$$

Applying three moments theorem, we get

$$M_A.l_1 + 2(l_1 + l_2)M_B + M_C.l_2 = \frac{w}{4}(l^3{}_1 + l^3{}_2)$$

$$\text{or } M_B = \frac{w}{4} \frac{(l^3{}_1 + l^3{}_2)}{(2l_1 + 3l_2)} \text{ lbs. ft.} \qquad (1)$$

Maximum positive moment in the middle span at centre

$$= \left(\frac{wl^2{}_2}{8} - M_B \right) \text{ lbs. ft.} \qquad (2)$$

Let M_X be the positive moment in the end span at any section XX′ at a distance X from A (Fig. 2)

$$\therefore M_X = \left(\frac{wl_1 x}{2} - \frac{wx^2}{2} \right) - M_B \frac{x}{l_1}$$

For maximum moment, differentiating the above equation we get

$$x = \frac{l_1}{2} - \frac{M_B}{wl_1} \qquad (3)$$

By substituting the value of x in the above equation of M_X, the value of maximum positive moment can be obtained.

To find out the reaction of the slab, we get

$$R_A = R_D \text{ and } R_B = R_C$$

$$\text{and } 2(R_A + R_B) = (2l_1 + l_2)\ W \text{ lbs.}$$

$$= 10.5\ W \text{ lbs.}$$

Now taking moments about B, we get

$$-M_B = R_A l_1 - \frac{wl^2_1}{2}$$

$$\text{or } R_A = \frac{Wl_1}{2} - \frac{M_B}{l_1} = Wx \text{ lbs.} \qquad (4)$$

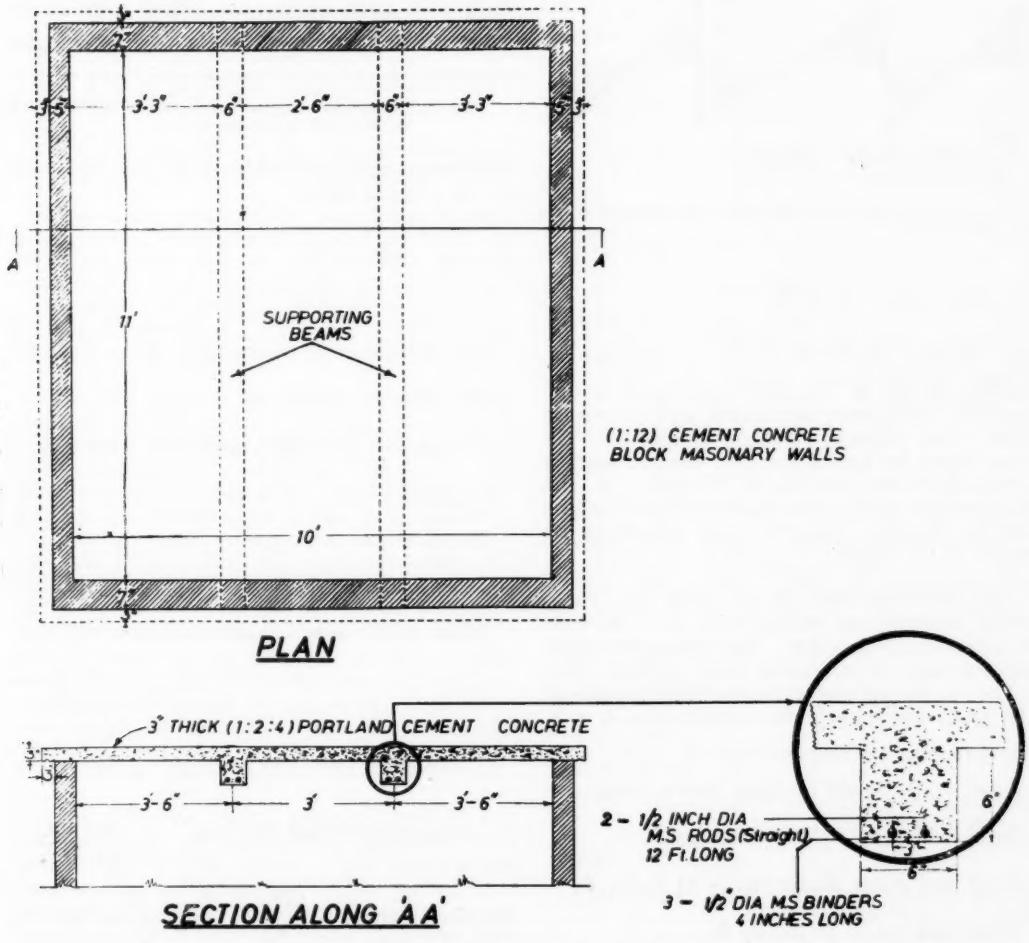

Fig. 1.—The constructional details of room with low-cost slab roof.

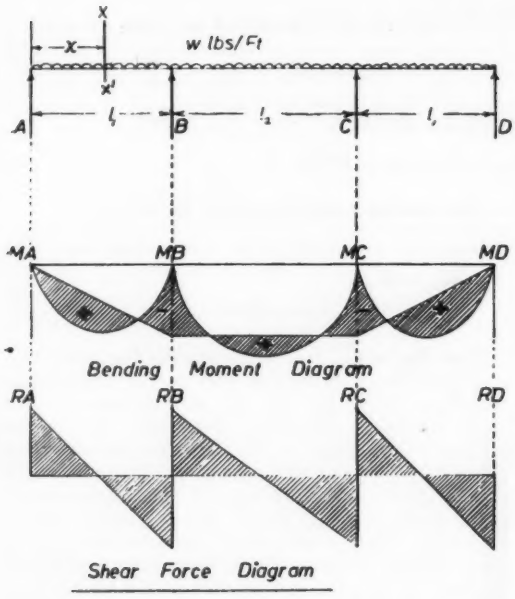

Fig. 2.—Bending moment and shear force diagrams of the slab. (Not to scale)

$$R_A + R_B = 5.25\,W$$

$$\therefore R_B = (5.25 - x)\,W \text{ lbs.}$$

The values of the bending moments and reactions have been calculated and shown in Table 1 for different values of l_1 and l_2. The actual spans for testing the roof were adopted as indicated in the last line of the table, as this arrangement gives equal maximum positive and negative bending moments with minimum reactions on the beams.

(*a*) *Slab.*—Let slab be 3″ thick of (1:2:4) cement concrete and plaster finish of ½″ thickness be given on both sides. The minimum superimposed load on inaccesible roofs has been specified as 120 lbs. per foot run in the British Standards. The minimum span under consideration is 3 ft., therefore, the superimposed load of $\frac{120}{3} = 40$ lbs. per sq. ft. should be taken into account.

Dead load of the slab = 36 lbs./sq. ft.

Dead load of the plaster etc. = 14 lbs./sq. ft.

Total load (w) = 90 lbs./sq. ft.

TABLE 1.—BENDING MOMENTS IN THE SLAB AND REACTIONS ON THE WALLS AND BEAMS.

Effective span l_1	Effective span l_2	Bending moment M_B (in. ft. lbs.)	Maximum positive moment in the end span (in ft. lbs).	x = Distance of the moment in column (5) from end A (in ft.)	Maximum positive moment in the middle span (in ft. lbs.)	R_A = R_D = Reactions on 5″ thick walls (in lbs).	R_B = R_C = Reactions on beams (in lbs.)
3′-0″	4′-6″	1.50W	0.50W	1.00	1.03W	1.00W	4.25W
3′-3″	4′-0″	1.32W	0.74W	1.22	0.68W	1.22W	4.03W
3′-6″	3′-6″	1.23W	0.98W	1.40	0.31W	1.40W	3.85W
3′-7½″	3′-3″	1.20W	1.10W	1.48	0.12W	1.48W	3.77W
3′-9″	3′-0″	1.20W	1.20W	1.55	-0.08W	1.55W	3.70W

NOTE :—The maximum negative and positive moments in the slab are equal in slab No. 5.

Maximum moment (Table 1) = 1.2 W lbs./ft. = 14.4 W lbs. inches

As $M = fZ = \frac{f\,bd^2}{6}$

$$\therefore f = \frac{6M}{bd^2} \quad \ldots\ldots\ldots\ldots \quad (b = 12'' \text{ \& } d = 3'')$$

= 0.8 W = 72 lbs./sq. in.

Therefore the thickness assumed is much safe.

(*b*) *Beams.*—

Effective span = 11.5 ft.

Dead load from slab = 3.70 × 36 = 133 lbs./Rft.

Dead load from beam projection 6″ × 6″ = 36 lbs.

169 lbs.

Total dead load 169 × 11.50 = 1940 lbs.

Minimum specified live load = 960 lbs.

2900 lbs.

Bending moment = $\frac{WL}{8}$ ft. lbs. = 50,000 inch lbs.

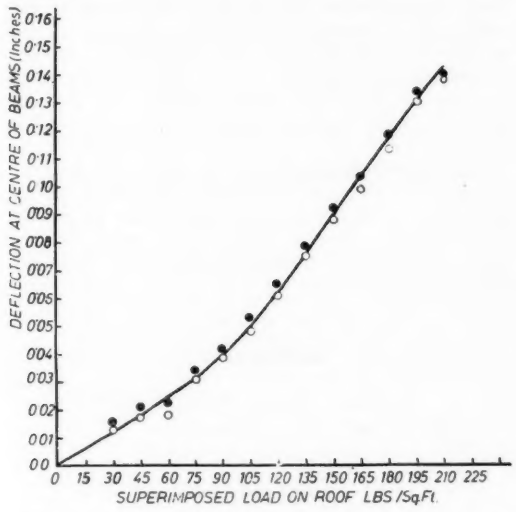

Fig. 3.—Observed relation between super imposed uniformly distributed load on the roof and deflection under beams at centre.

Taking the beam as of rectangular section, the stresses in steel 18,000 lbs. per sq. inch, the maximum stress in concrete comes to 760 lbs./sq. inch. Two bars of ½″ dia. are found sufficient. The beam is safe in shear and bond stresses.

Testing under Uniformly Distributed Load

The roof was laid in (1:2:4) cement, Malir sand and double screened coarse aggregate (Fig. 1) from Malir River bed. The water cement ratio (which was inclusive of the water absorbed by the dry aggregate) was kept at 0.6, as this is generally specified on Government works. The centering was kept in place for the normal period and then removed. The ceiling and roof were then plastered with (1:6) cement-sand mortar. After 56 days, the roof was progressively loaded with sand bags as shown in Table 2. The superimposed load was increased by 15 lbs. after every 48 hours except in two cases when the superimposed load were kept unchanged for 72 hours. Deflections were recorded under the beams at centre and plotted against loads (Fig. 3). These tests were conducted by Mr. Mubarak Ahmad Choudhari.

The deflection gauges were removed at 210 lbs. load and the beams were supported with wooden planks from below. The superimposed load was again increased by 15 lbs. per sq. ft. of the roof after every 48 hours. Cracks a ppeared approximately at the point where maximum positive moment occurs, and were first noted at a live

TABLE 2.—DEFLECTION TESTING OF THE DESIGNED ROOF UNDER VARYING SUPERIMPOSED LOAD.

Date	Total superimposed load in lbs./sq.ft.	Deflection at centre in inches		Remarks
		Beam No. 1	Beam No. 2	
8.10.60	30	.000	.000	The readings of deflection were taken before increasing the loading of the roof to the next value.
10.10.60	30	.008	.010	
11.10.60	45	.015	.013	
12.10.60	45	.016	.013	
13.10.60	60	.021	.017	
14.10.60	60	.022	.018	
15.10.60	75	.022	.018	
16.10.60	75	.026	.022	
18.10.60	90	.034	.031	The superimposed load of 75 lbs./sq. ft. was allowed to remain for 72 hours.
19.10.60	90	.036	.033	
20.10.60	105	.042	.039	
21.10.60	105	.046	.042	
22.10.60	120	.053	.048	
23.10.60	120	.054	.050	A horizontal crack appeared all round in the supporting walls at about 9″ below the ceiling level.
24.10.60	135	.065	.061	
25.10.60	135	.063	.064	
26.10.60	150	.079	.075	
27.10.60	150	.085	.081	
29.10.60	165	.092	.088	The superimposed load of 150 lbs./sq. ft. was all wed to remain for 72 hours.
30.10.60	165	.095	.090	
31.10.60	180	.103	.099	
1.11.60	180	.108	.104	
2.11.60	195	.118	.113	
3.11.60	195	.120	.115	Fine cracks appeared in the middle of the beams.
4.11.60	210	.133	.130	
5.11.60	210	.140	.138	The defleetion was noted after 24 hours of 210 lbs. superimposed load.

load of 255 lbs./sq. ft. The loading on the roof was continued till 360 lbs./sq. ft. was reached., At this load, the cracks slightly increased in length. As further loading was not possible, the load of 360 lbs./sq. ft. was maintained for about a month. The rainfall in the meantime also increased the live load, but the roof was unaffected. When the loads were removed, there were no cracks observable on the top of the slab.

Conclusion

The test of the roof has shown that the flexural strength of the concrete can be successfully used to minimize the use of the steel to a very considerable extent in the roof construction. The high test load also indicates that the type of roofing described in this paper can be used in domestic houses of more than one storey. If the flexural strength can be relied upon to a geater extent than is normally assumed, then further economy in steel is possible by giving only one beam at the centre of the rooms. Further experiments aimed at studying the flexural and compressive properties of the concrete based on locally manufactured cement are therefore being carried out, and the results will be reported separately.

Acknowledgement.—The author is grateful to several colleagues for helpful discussions in the problem, to Mr. Mahmood Akhtar for help with some of the constructional work, and to Mr. Mubarak Ahmad Choudhari for help with the testing of the roof.

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UTILISATION OF 'BALCRETE' FOAMING AGENT

Part I.—Production of Cellular Concrete (with Neat Cement)

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(Received January 5, 1961)

This paper deals with the preparation and physical properties of moist cured cellular concrete based on neat cement. Methods of production based on high speed stirrers and ordinary non-tilting drum concrete mixer using 'Balcrete' foaming agent have been successfully developed. The ratio of rate of production to mixer capacity is 2.6-2.8 which is very satisfactory since the highest reported ratio using assembly line techniques is 3.0. Compressive and flexural strengths as well as thermal conductivity values have been determined for cellular concrete of varying densities. The ratio of flexural to compressive strength varies from 0.12 to 0.20, the ratio increasing with increase in density. This is in contrast to the results given in the literature for autoclaved cellular concrete in which case the ratio decreases with increase in density.

The cellular concrete has adequate strength for structural use at densities above 60 lbs. per cu. ft., for partition walls at densities between 40-60 lbs. per cu. ft., and for thermal insulation at densities below 30 lbs. per cu. ft.

Introduction

Concrete weighing from 20 to 100 pounds per cubic foot and having a homogenous cell structure is usually known as cellular concrete although descriptions like 'aerated', 'porous' and 'foamed' concrete have been used, irrespective of the method of producing the cell structure. These definitions do not ordinarily encompass light-weight aggregate concretes.

Various methods in use for the production of cellular concrete may be classified as follows:—

(i) Processes in which a gas is produced by a chemical reaction within the mix before it sets.

(ii) Processes involving use of excess water which on drying out leaves air-filled pores. This method of making cellular concrete was first patented in 1930,[1] but has found only limited use and that also in conjunction with the aeration by chemical means.

(iii) Foaming processes, which may be described as air-entraining in character, the additives used for the purpose being known as 'foaming' or 'air-entraining agents'. This type of process was first patented by Bayer[2] in 1923. The following types of foaming agents have been used:

(a) Detergents (sodium lauryl sulphate, alkyl aryl sulphonate etc.).

(b) Resins, oils and their soaps.

(c) Hydrolysed protiens containing suitable additives to provide stable foam volume.

Agents in category (c) have come to be used extensively for cellular concrete in recent years. A composition for foaming agent belonging to this class and developed at these laboratories[3] has been used in the investigations reported in this paper.

The paper is confined to cellular concrete subject to moist curing since the alternative method of curing in autoclaves at about 350 °F. was not considered practical or economical for this country at this stage. The present study may be divided into three steps, namely (a) preparation of cellular concrete, (b) control of density of cellular concrete and (c) examination of physical properties of cellular concrete.

Preparation of Cellular Concrete

Two types of methods are commonly used for production of cellualr concrete: (a) 'Mix-foam' method in whch foaming agent is added to the mixture after all the other ingredients have been mixed to a fairly thin slurry. High speed stirrers (1400-2800 r.p.m.) or concrete mixers with motor attachments to permit continuous variation in paddle speed from 30 to 90 r.p.m. are recommended for this method. (b) 'Pre-foam' method which involves the generation of foam by aerating a protein base compound through a nozzle and adding it to the concrete in place of the usual aggregates. Several variations of these methods have been investigated in the present study.

(a) High speed stirring mixer (Fig. 1): For laboratory work high speed turbo stirrer (A Gallenkamp & Co. Cat. No. 35-705) using stirring rotor turbo reactor has been used for investigations but

for regular production a similar stirrer of diameter 14" with a speed of 1425 r.p.m. using 7.5 H.P. motor was fabricated and used for the purpose. This particular stirrer has been used for producing 40,000 cu. ft. of cellular concrete from neat cement (density 30-35 lbs./cu. ft.) at Chichoki Hydel Station. The method of using this stirrer is as follows:

Three and a half gallons of water is taken in 45-gallon barrel and 56 lbs. of cement added gradually with continuous agitation. When the cement and water slurry are thoroughly mixed, one litre of foaming agent is added and the mixing continued for 7-10 minutes. Finally, the material is drawn off from the drum and poured into moulds for making bricks. Part of the production at Chichoki was cast in situ on a flat roof. For this purpose the roof area was divided into 2′ × 4′ panels using wooden forms into which foam concrete was poured to a depth of 3". Alternate panels were poured together so that as every other section sets, the forms could be removed and the intervening area filled.

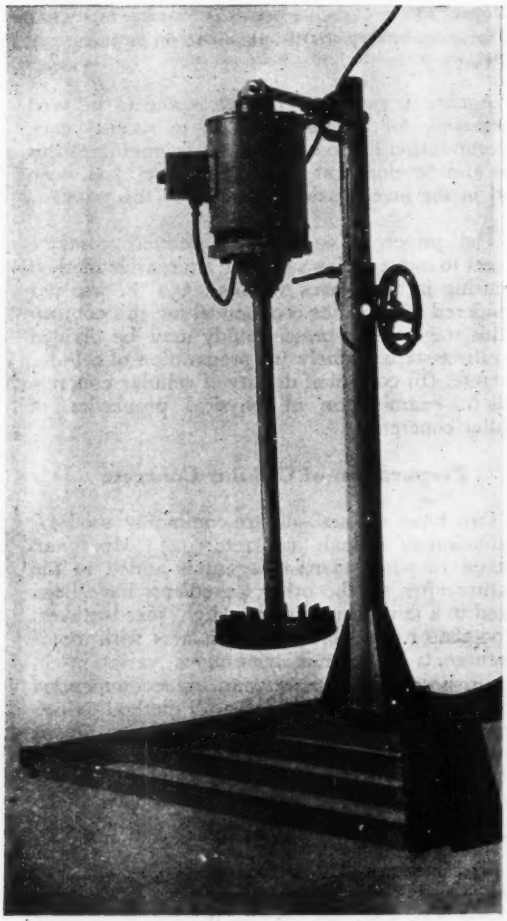


Fig. 1

Control of density of cellular concrete prepared by this method is comparatively easy, but this stirrer is useful only for neat cement and failed to give satisfactory results when sand had to be incorporated in the mix.

(b) Cement mixer, tilting drum type capacity 2 cu. ft. (Fig. 2). This mixer has been modified to revolve at a speed of 40-50 r.p.m. The details of producing cellular concrete by this method are similar to those described for the high speed stirrer. Attempts to generate foam in the mix before adding cement did not give satisfactory products, but it is believed possible to get better results, if the mixer is run at a low speed in the initial stages. This method has one advantage over the high speed stirrer method, namely that cellular concrete of all types can be produced, whether containing neat cement or mixtures of cement and aggregate.

(c) Ordinary non-tilting drum concrete mixer-capacity 6 cu. ft. Speed of revolution 10-12 r.p.m. (Fig .3) This type of mixer is easily available in the country, and it was therefore considered desirable

Fig. 2

to work out a method for production of cellular concrete without any modifications of the normal mixer. The method which has been found to work most satisfactorily with this mixer is described below.

Fifteen gallons of water and 4.5 lbs. foaming agent (2.0% by weight of cement) are poured in the mixer, which is then run for 2-3 minutes so as to generate foam. 224 lbs. of cement is gradually added to the foam. The mixer is run for about fifteen minutes altogether, when a uniform slurry is obtained, which is drawn off from the mixer and poured into wooden moulds. The moulds are opened after 18-24 hours to avoid breakages.

This mixer gives satisfactory results even when aggregate is used as one of the ingredients, and has been used by the Pakistan P. W. D., Lahore, for P. & T. 'Repeater huts' for production of cellular concrete containing cement and sand in the proportions of 1:1 and 1:3.

The quantity of foaming agent required for producing 100 cu. ft. of cellular concrete of density 30-35 lbs./cu. ft. based on neat cement is given in Table 1 for the three methods (a), (b) and (c), while Table 2 gives the corresponding rates of production.

TABLE 1.—EFFECT OF METHOD OF PRODUCTION ON FOAMING AGENT REQUIREMENT.

Method of production	Amount of foamig agent	
	by weight of cement %	per 100 cu. ft. of concrete
(a)	3.5	140 lbs.
(b)	3.0	120 lbs.
(c)	2.5	100 lbs.

TABLE 2

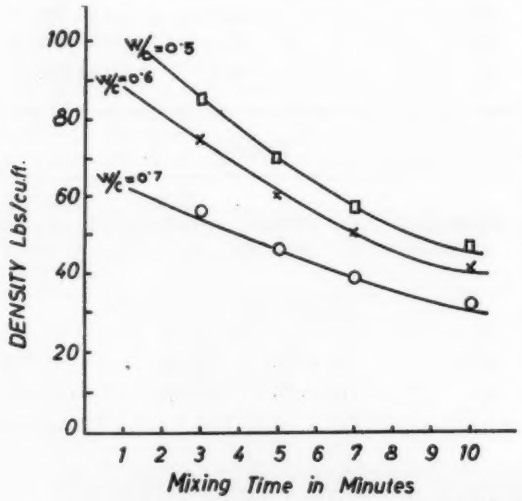
Method of production	Mixer capacity	Rate of production cu. ft./hr.	Rate of production to mixer capacity
(a)		6- 7	
(b)	2 cu. ft.	4- 5	2-2.25
(c)	6 cu. ft.	16-17	2.6-2.8

Fig. 3

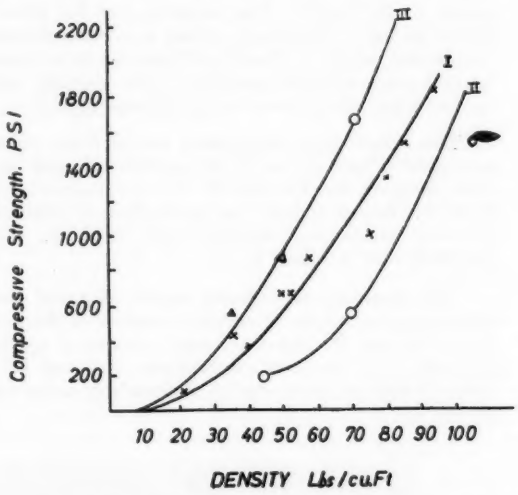
According to National Foam System Inc., Pa., U.S.A ,[4] their preformed foam method can use 'assembly line techniques' in making installation, mixing and pouring 15 cu. yd. of concrete an hour using a 5 cu. yd. end loaded transit mixer. The ratio of hourly production rate to the mixer capacity in this case works out to 3.0 whereas for the method (c) described above without the use of any assembly line techniques, the ratio of production rate to mixer capacity is 2.6-2.8 which is very satisfactory indeed.

Control of Density of Cellular Concrete

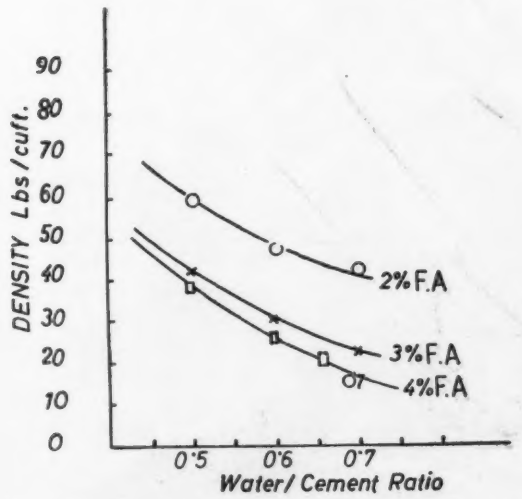
Since most of the physical properties of light-weight concretes depend quite sensitively upon



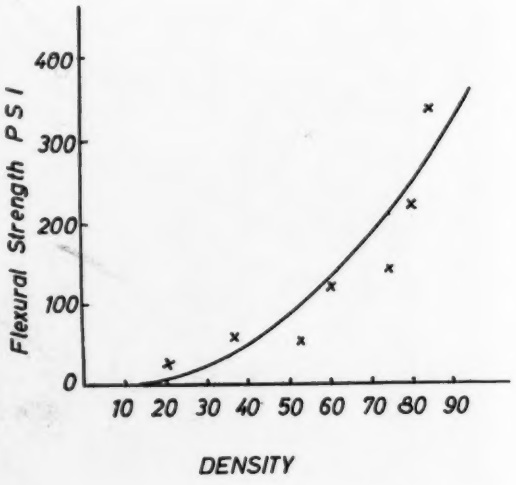
Graph 1.—Effect of variation in mixing time and water/cement ratio on density of cellular concrete using high speed laboratory mixer.



Graph 3.—Relationship between compressive strength and density for: (i) moist cured cellular concrete (CSIR), (ii) light-weight aggregate concrete, and (iii) autoclaved cellular concrete.

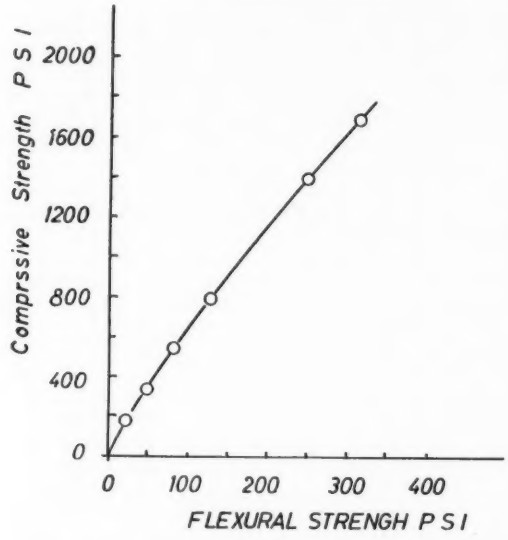


Graph 2.—Effect of variation in proportion of foaming agent used and water/cement ratio on density of cellular concrete using ordinary non-tilting drum concrete mixer.

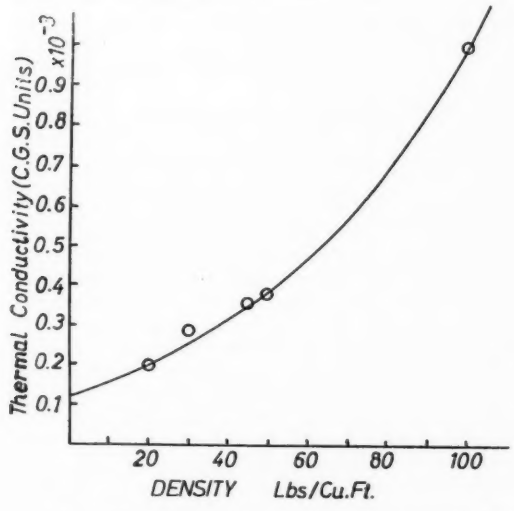


Graph 4.—Relationship between flexural strength and density for moist cured cellular concrete.

density, it is important to have a fairly rigid control on density of the product, which depends on several factors, viz., quantity of foaming agent, type of mixer used, length of the mixing cycle and the water cement ratio. The effect of mixing time and water cement (W/C) ratio on the density of the cellular concrete using 3% foaming agent by weight of cement and high speed laboratory stirrer is shown in Graph 1. Control of density using an ordinary cement mixer is possible by adjustment of the quantity of foaming agent and the water/cement ratio, since minimum mixing time had to be maintained at 15 minutes to get a uniform slurry. The results are shown in Graph 2. Cellular concrete of densities below 30 gave depression in precast blocks on curing and it was therefore found necessary to use calcium chloride 1-2% by weight of cement. In case of method (1) and (2) calcium chloride is added to water in the very beginning, whereas in case of method (3) calcium chloride is added during the last five minutes of the mixing time.



Graph 5.—Relationship between compressive and flexural strength of moist cured cellular concrete.



Graph 6.—Relationship between thermal conductivity and density of moist cured cellular concrete.

Physical Tests on the Cellular Concrete

Bricks made for testing the physical properties have been moist cured and strengths reported are for specimens 28 days old and tested in a room dry condition.

The compressive strength was determined on 4″ cubes in a 40-ton hand operated compression testing machine. The relationship between density and compressive strength is shown in Graph 3 and for comparison, curves for light weight aggregate (pumice) concrete and autoclaved cellular concrete as reported in the literature[5] have been included in the graph. For the flexural strength, 18″ × 4″ × 4″ bricks were tested at a span of 15″, and the results were calculated according to ASTM specification C 293-54 T (1955). The relationship between density and flexural strength is shown in Graph 4 and that of compressive with flexural strength in Graph 5. The ratio of flexural to compressive strength varies from 0.12 to 0.20, the ratio increasing with increase in density. This is in contrast to the results given in literature for autoclaved cellular concrete in which case this ratio decreases with increase in density, the ratio varying from 0.35-0.20.

The thermal conductivity values for moist cured cellular concrete made of neat cement are given in Graph 6. The method used for determining the coefficient of thermal conductivity was that described previously by Ahsanullah, Ahmad and Chotani.[6] K value for local cement brick as determined by the method is 0.68 ± 0.03 cal. cm. sec.$^{-1}$/°C., whereas values reported for cement in the literature vary from 0.8-1.2. This together with the earlier anomaly in the ratio of flexural to compressive strength suggests the need for a fuller investigation of the cement as such.

The properties of moist cured cellular concrete containing portland cement with sand and other aggregates and the economics of production of

various types of cellular concrete will form the subject matter of Part II of the present paper.

Conclusions

Whereas cellular concrete has been produced commercially in Europe for over 30 years and its use in the U.K. and the U.S.A. has been comparatively recent, in Pakistan, the C. S. I. R. has been the first to introduce cellular concrete in the country and develop an economical and easily controlled method of its production. On the basis of the foregoing results in this paper, it may be recommended that cellular concrete of lower densities in the 30 pounds per cubic foot range may be used where insulation value rather than structural strength is the determining factor. For structural applications the density range that may be recommended is from 60 pounds per cubic foot up. The lower densities (45-60) are useful for partition walls. Cellular concrete may als be used as roof or floor fills and for fire protection.

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A GRAVITY SURVEY OF QUETTA AND MASTUNG VALLEYS*

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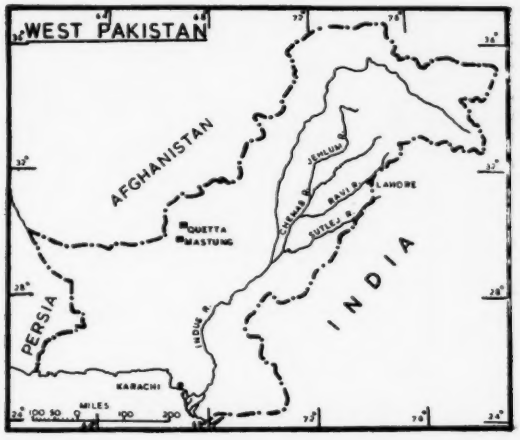
The gravity survey started by Moiduddin and Thirlaway in Khad Kucha, south of Mastung, has been extended into the Mastung and Quetta valleys. It is thought that large scale faulting was a primary factor in the origin of the valleys and this is supported by the gravity data. Of particular interest are cross faults which must have modified the early drainage and have been a major factor controlling the character of ground-water aquifers.

There is some confirmation for Moiduddin and Thirlaway's hypothesis that the observed differences in ground water resources within the alluvial materials may depend on the materials having accumulated in either a lake or in a river system. Most of the material filling the valleys is probably of Siwalik age.

1. General

Recently, Moiduddin and Thirlaway investigated the groundwater resources of the Khad Kucha valley lying immediately south of Mastung, by using a combination of gravity and seismic refraction techniques. The interpretation, supported by evidence from two test holes, indicated the presence of a maximum of 2500 feet of low density rocks deposited on the basement rock of the valley. This raised questions as to the history and origin of these deposits. The immediate practical problem was whether, or not, the methods used could determine the history and origin of the deposits with sufficient precision to enable the total groundwater resources of the mountain valleys to be predicted. The present paper extends this inquiry into the Quetta and the Mastung valleys proper. The location of these places is shown in Map 1. The geology of the Mastung valley has already been described by Moiduddin and Thirlaway.[1]

Geology of the Quetta Valley.—The geology of the Quetta valley is similar to that of the Mastung valley. The surrounding hills are of tertiary and mesozoic limestones and shales, with a steep westerly dip. The trend of the hills, generally, marks the strike direction. The valley is filled with horizontal quaternary and recent deposits. A large number of test holes were drilled for water at various places in the valley and encountered clays and sands interbedded with gravels. None of these test holes reached the basement. The deepest was 1223 feet, near village Kasi (Map III), and at a distance of 2500 feet from the nearest limestone outcrop. Down to 531 feet the formations consisted of limestone boulders, gravels and clays. Beneath this occurred red clays. Another well in the St. Mary's churchyard, Quetta,



Map I.—Map of West Pakistan showing the location of the areas surveyed.

met red clays at 344 feet after passing through recent gravels. The red clays are thought to be Siwalik in age.

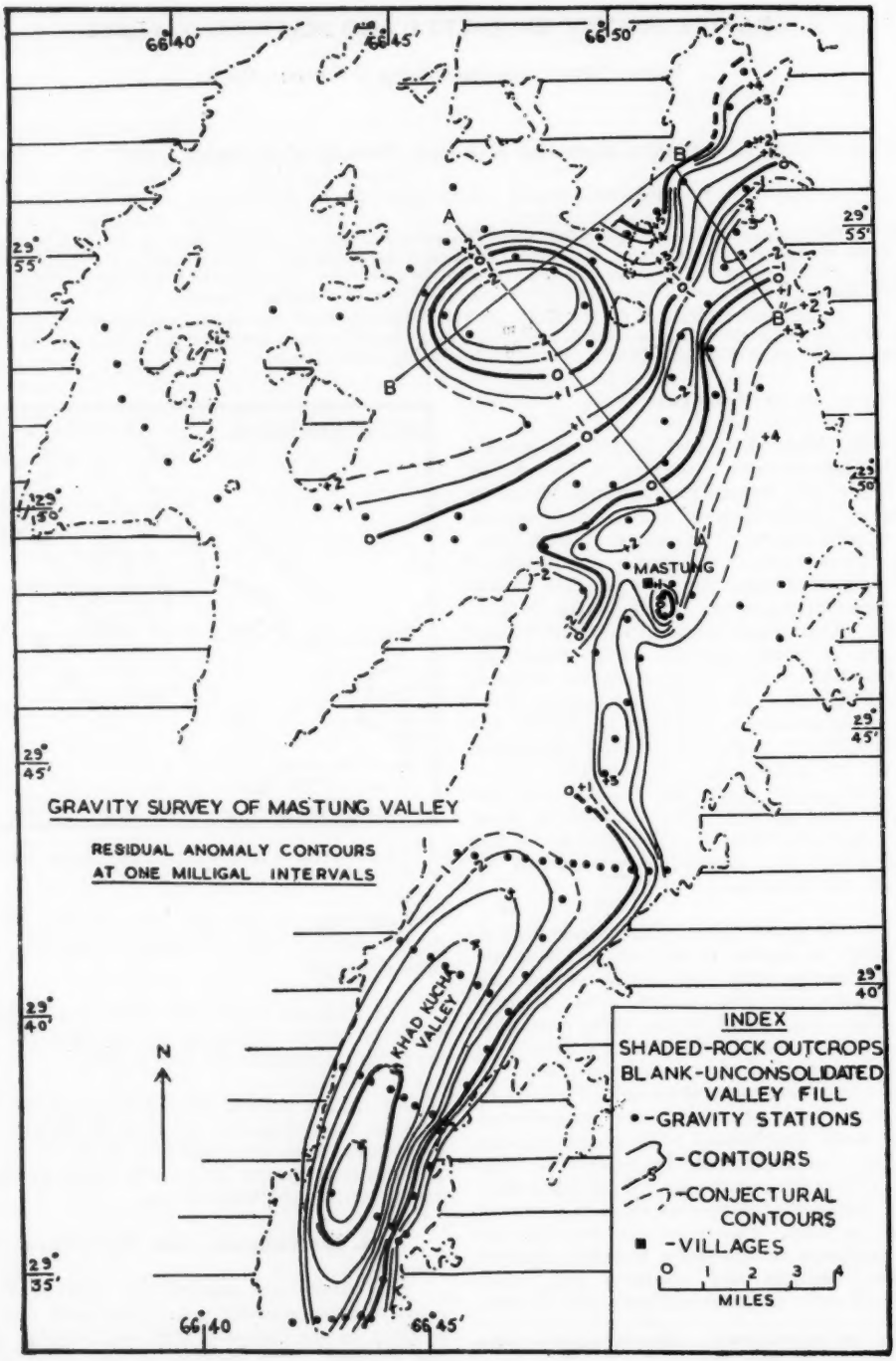
The majority of the test drills met with good yields of water, averaging 5000 gallons per hour from the unconsolidated gravel beds.

The mean annual rainfall is about $9\frac{3}{4}$", and only the main drainage channel, the Sariab Lora, is perennial with a normal flow of about 2 cusecs. The drainage system is towards north, eventually joining with the Pishin Lora.

2. Instruments and Procedure

The survey was carried out with a Worden gravity meter number 372. The scale constant, as given by the manufacturer, was 0.09844 mgal/dial division. The instrument was read at the

*Read at the Section Meeting, Eleventh Pakistan Science Conference, Karachi, February 1959.



Map II.—Gravity map of the Mastung valley.

base station in the morning and in the evening of each field day. Field observations were reduced to the base under the assumption that the drift remained linear during the day.

Heights were measured using a barometric altimeter with a scale constant of 10 feet/division. The diurnal variations of barometric pressure, as recorded at the geophysical institute in Quetta, were used for corrections. Bench mark heights were used, wherever available. The standard deviation of heights obtained in this manner was $\pm$ 5 feet. A bench mark in the geophysical institute was selected as the main base. A sub-base was chosen in the District Board Office in Mastung town for the Mastung valley survey.

Field stations were placed, in general, a mile apart along the main roads. A few stations were situated off the roads and had to be reached by foot. Positions were taken from the topographic maps of the Survey of Pakistan (scale 1 inch to 1 mile). Distances between field stations and the base are correct within 300 feet.

Reduction of Field Data.—The field data were reduced, in the normal manner, for difference in latitude and height, and for the effect of visible masses. From the international gravity formula[2], latitude correction factors of 1.149 mgal/mile for the Quetta valley and 1.133 mgal/mile for the Mastung valley were adopted. The density was assumed to be 2.67 g./cm.^{3}, and this gave a height correction of + 0.05998 mgal/foot.

Terrain corrections were calculated after King Hubbert,[3] who gives the effects of 47 common topographic features. The terrain correction magnitudes were calculated along several topographic cross-sections plotted from the one-inch maps of the areas in question. A terrain correction map constituting "equi-terrain correction" contour lines and representing this correction as an areal function was obtained in this way. Thus the location of a field station on this map was sufficient for determining the necessary terrain correction.

3. Sources of Error

Systematic Errors.— These could be introduced by an incorrect calibration of the gravity meter. According to Woollard,[4] the calibration of Worden gravity meters is correct to about 1 part in 1000 to 2000. The maximum gravity change being only 30 mgal, the systematic error due to calibration is smaller than 0.03 mgal and can be neglected.

Another source of systematic errors is the elevation correction. The density for the Bouguer correction was taken as 2.67, which might have been too high for most of the stations in the valleys. The boreholes show a predominance of clays and gravels in the overburden, which would indicate a density of about 2.3 g./cm.^{3} The maximum height difference in the field survey was 500 feet, giving rise to a maximum error of about 3.5 mgal due to this factor. This systematic error, which has a positive correlation to the topography, does not change the overall gravity picture seriously and therefore has but little effect on the broad interpretation.

Random Errors.—The main cause of random errors was the limited accuracy of heights. As stated above, the standard deviation of altimeter heights was $\pm$ 5 feet, introducing an error of $\pm$ 0.3 mgal in the elevation correction. The latitudes are supposed to be correct within 300 feet, therefore the maximum error in the latitude correction should be $\pm$ 0.07 mgal. Errors in the reading of the gravity meter were of the order of $\pm$ 0.02 mgal, while errors due to the assumption of a linear drift curve might have been as much as 0.05 mgal.

In preparing the terrain correction maps, the actual 3-dimensional topographic features were reduced, for simplicity, to 2-dimensional features. In addition to this, the more or less irregular topographic sections had to be smoothened for matching them with the geometric features considered in Hubbert's work. Since the length of the mountains dealt with, is several times greater than their width, these simplifications are justified and have not introduced gross errors as regards interpretation.

The random error from all these sources is estimated to be about $\pm$ 0.5 mgals. Since the total gravity variation in the two valleys is about 30 mgals, and 1-mgal contours are presented, the anomalies shown are real.

4. Interpretation of Results

General.—The gravity map was prepared on the basis of residual anomalies, and has been corrected for all the factors influencing gravity measurements, except density variations below datum level. It represents, therefore, a picture of the density variations in the subsufrace. Sharp gravity gradients which can be caused only by an abrupt change of density of the subsurface materials are interpreted as faulted structures and gravity troughs as deep basins filled with lighter materials. These are the fundamental principles used in interpretation of this survey. Estimates of thickness of low density materials which are thought to cause the ano-

malies depend on the expression for the attraction of an infinite slab. Numerically this reduces to

$$\frac{78}{\Delta \rho} \times \Delta g = h$$

$\Delta \rho$ being the density contrast between the materials constituting the overburden and the basement rock.

Any given gravity field can be produced by an infinite number of mass distributions.[5] Therefore, an interpretation of gravity map cannot be unique. This means that, in the absence of additional information, no definite conclusions can be drawn from a gravity map. It is, however, possible to calculate the maximum depth for the case of a given gravity anomaly.

The interpretation of the gravity anomalies presented in this paper has been carried out along qualitative lines by using the gravity gradients along the sections indicated on Maps II and III, and assuming a difference in density of 0.4 g./cm.^{3} between the valley fill and the solid rocks. The given geometric form of the subsurface should be regarded as a first approximation only, and modifications may be necessary on detailed analysis. The major features, however, are likely to remain as described below.

Mastung Valley.—The gravity anomalies of the Khad Kucha and Mastung valleys are shown in Map II. The gravity values increase northwards in the Khad Kucha valley to the vicinity of Mastung, where the pattern changes. In the Mastung valley there are no pronounced variations in gravity, and particularly noticeable is the absence of any marked gravity "lows" comparable with that in the Khad Kucha valley.

Near Mastung, however, there are some short but high gradients which have been interpreted as marking a cross-fault which separates the two valleys. At the times no data was obtained from the area about 4 miles southwest of Mastung, but investigations now being carried out by Mr. Zakauddin of the Geophysical Institute have revealed the presence of a gravity high in this zone. This follows the tendency of the contours shown, and substantiates the picture of the Mastung valley as a structural high relative to the Khad Kucha valley.

The high gradient along the section B′ B″ (Fig. 1a) is interpreted as due to a fault. Similarly, the relatively high gradients on the eastern boundary of the valley are thought to be due to strike faults.

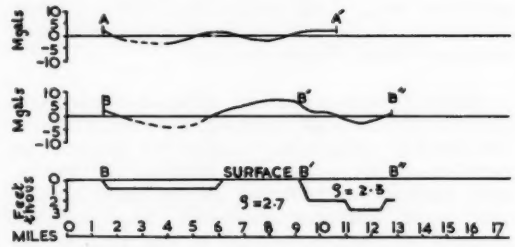


Fig. 1(a) —Gravity profile of the Mastung valley.

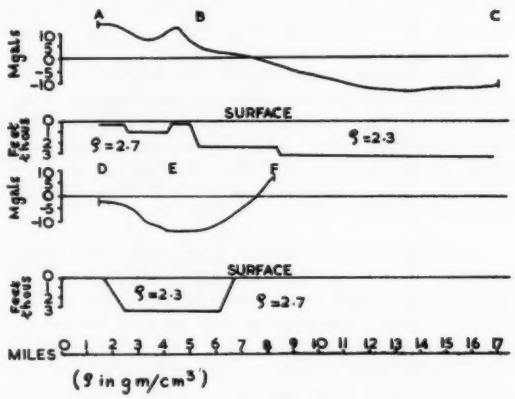


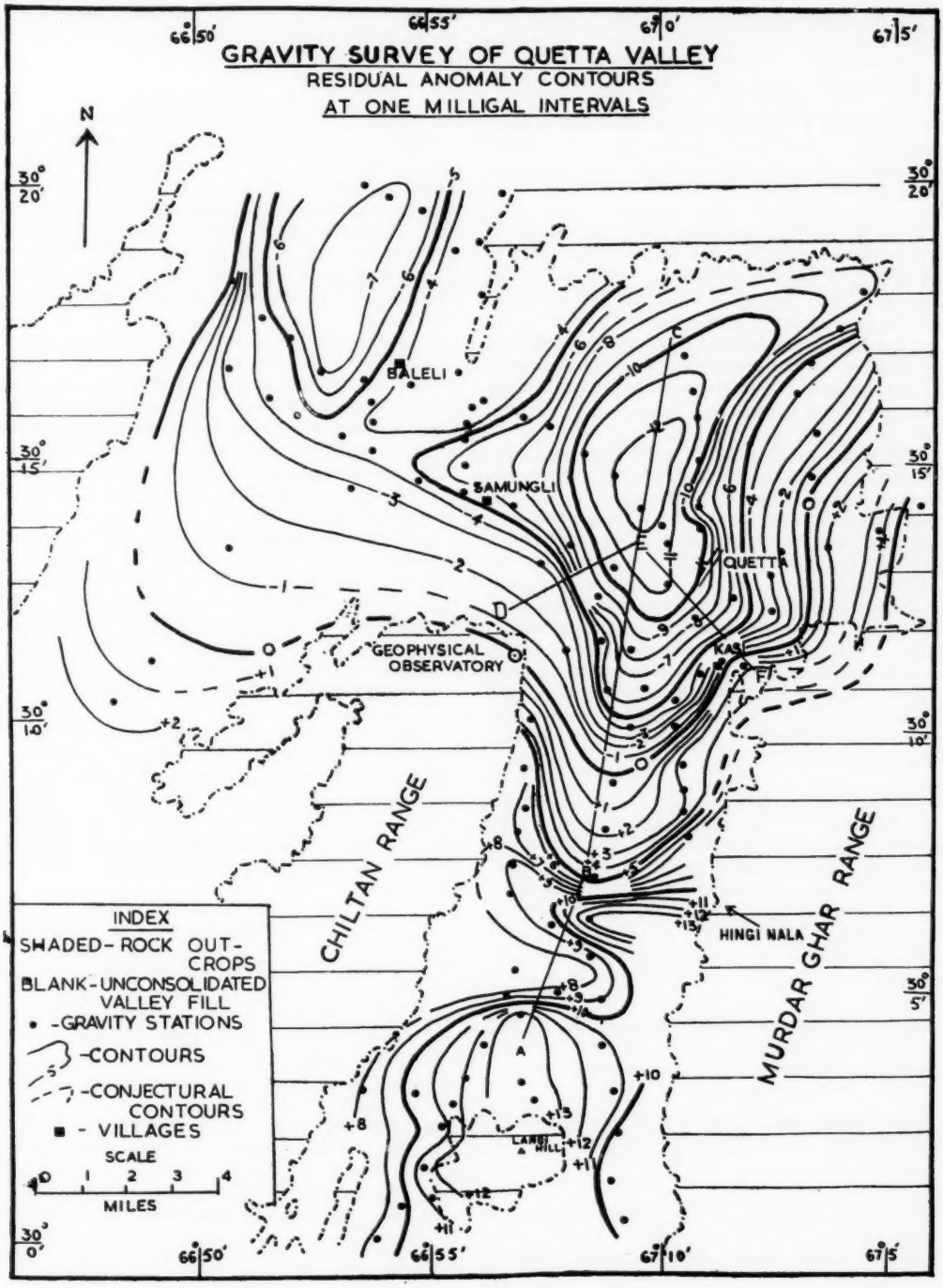
Fig. 1(b) —Gravity profile of the Quetta valley.

The interpreted shape of the subsurface is shown in each case under the gravity profile.

The gravity anomalies of the major part of the Mastung valley are very small, and show no marked pattern. The cross-section along BB′ does not indicate any major structural or erosional features. Unfortunately, insufficient data was obtained from the western and northern parts of the valley to justify drawing gravity contours. Mr. Zakauddin's investigations in these parts of the valley as well as in the Aghberg valley north of Mastung have revealed rapidly rising gravity anomalies.

It appears, therefore, that the central portion of the Mastung valley and the related Khad Kucha valley are structurally lower than the area to the northwest, dropping in the direction of the present drainage. They are bounded by major northerly striking faults and separated from one another by cross faults. This gives some confirmation to the idea that earlier the Khad Kucha valley was separated structurally from the Mastung valley, and may well have been the site of a lake.

Quetta Valley.—The most prominent feature of the gravity map of Quetta valley (Map III) is a



Map III.—Gravity map of the Quetta valley.

big gravity basin with its centre 3 miles northwest of Quetta City. This basin is bounded by sharp gravity gradients. The relatively low gravity anomalies continue as far south as the point where the Hingi Nala enters the valley. Then there is a sharp gradient marking a 'spur-like' high, about a mile wide. Still further southwards the increasing gravity anomaly with a sharp gradient encircles Landi hill.

West of the big gravity basin, a trough of gravity low runs about 11 miles from Quetta, after which it turns northwards. The minimum thickness of low density material in this trough is 1000 feet assuming a density difference of about 0.4 g/cm.^{3}. The resistivity observations made by Mr. Hashim of the Geological Survey indicated 600 feet, at least, of uniform alluvial material in the centre of this trough.

The main gravity "lows" are interpreted as deep structural basins filled with light materials. The zone of low values of gravity running through the Baleli Gap is thought to mark a buried channel. The main channel appears to be obstructed about 7/8 miles south of Quetta. The gravity gradient is very sharp here – about 8 mgals/mile – and indicates the presence of a cross-fault. About 2 miles southwards, the gradient again becomes sharp — about 6 mgals/mile — and can be explained in the same way. High positive anomalies in the extreme south are due to the rapidly decreasing depth of the basement rock which can be seen cropping out at two places in the middle of the valley.

The gravity profiles along two cross-sections *viz.* ABC and DEF are shown in Fig. 1 (b). The interpretation of the profiles has been done from an examination of the gradients. The approximate shape of the subsurface structure is calculated after Shaw's method.[6] In these calculations, a fill of average density of 2.3 g./cm.^{3} overlying a basement rock of density 2.7 g./cm.^{3} has been assumed.

Although the cross-section ABC is interpreted as caused by a major cross-fault, the gradient variations could be due to erosional features.

The interpreted section corresponding to profile DEF indicates strike faulting along both sides of the valley.

The evidence from surface geology in support of the interpretation of the gravity data is available at certain places. A fault can be observed in the outcrop near the place where the Hingi Nallah enters the valley. Another faulted zone is seen at the border of the valley formed by the Chiltan range.

5. Conclusions

Both the Quetta and Mastung valleys are primarily structural features modified by erosional agents at later stages. A maximum of about 2500 feet of low density materials have accumulated in the Khad Kucha valley compared with about 4000 feet in the Quetta valley. The importance of cross faults as well as strike faults in the genesis of these valleys has been emphasised by the gravity survey.

Knowledge of the history and origin of these valleys is essential to an understanding of the groundwater resources. This work has lent further support to the idea that recent deposits have accumulated under lake conditions in Khad Kucha, while drainage in Quetta found free outlet through the Baleli Gap Channel.

Acknowledgements.—The authors are greatly indebted to Dr. H. I. S. Thirlaway for suggesting this work, and giving valuable guidance in the field. The senior author wishes to express his thanks to the Director of the Geological Survey, Dr. E. R. Gee., and Mr. A. M. Khan for allowing him to undertake the work; and to Mr. Zakauddin for help with the computations.

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PRELIMINARY SURVEY AND CHEMICAL INVESTIGATIONS ON IRON ORES OF KHYBER AGENCY

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(Received October 4, 1960)

The authors have surveyed and chemically investigated the iron ores of Khyber Agency. Ore from Lashoora, a place eight miles from Jamrud, was found to be relatively rich in iron content but had a large percentage of silica (22.9% free and 8.5% combined) which makes it difficult to reduce. Attempts were made to reduce the silica content by washing, and gravity separation. The results are not very encouraging. The use of a magnet for concentrating the iron oxide particles has also failed. It is suggested that ore from greater depth be studied and the possibility of the production of ferrosilica examined.

Deposits of iron ore have been reported from time to time in the various regions of Khyber Agency, and some fantastic claims have been made about the quality and the extent of such deposits, but so far no systematic geological or mineralogical studies have been reported. The present investigations have been undertaken specially with the purpose of assessing the qualities of iron deposits in several areas in Khyber Agency. The difficulty of transport in the hilly areas and the non-availability of good quality metallurgical coke complicate the problem.

1. Survey

The areas surveyed are the following: (vide Fig. 1)

1. Lashoora, situated at about 8 miles from Jamrud 300° north-west and about four miles from Gudar 330° north-west.

2. Shilman, situated at about 10 miles north-east of Landikotal.

3. Jabba, situated at 10 miles from Jamrud in the direction of 290° south-west and 1½ miles 33° north-west from Tanga.

4. Tanga, situated at 7 miles 21° south-west from Jamrud.

A. Lashoora

The colour of the rocks is black, grey and brown due to the intermixture of light and dark minerals. The rocks are folded and consist of shaly and slaty formations. The rocks are also crumpled and show signs of weathering and erosior Quartzite bands are innumerable and occur in veins varying from ¼" to 6" in width.

An outcrop of hematite exists at about 1500 ft. altitude. The outcrop was 38 ft. in length, 8 to 10 ft. in width and 10 ft. in depth. The outcrop vanishes and again crops out at a distance of 200 ft. in the same continuity. It was varying in width from 2 to 3 feet and had a length of 25 ft. estimated after digging at certain intervals. The total outcrop of hematite exposed on the surface is about 50 ft.in length. The exact depth could not be measured due to lack of facilities of hard drilling; the maximum depth that could be

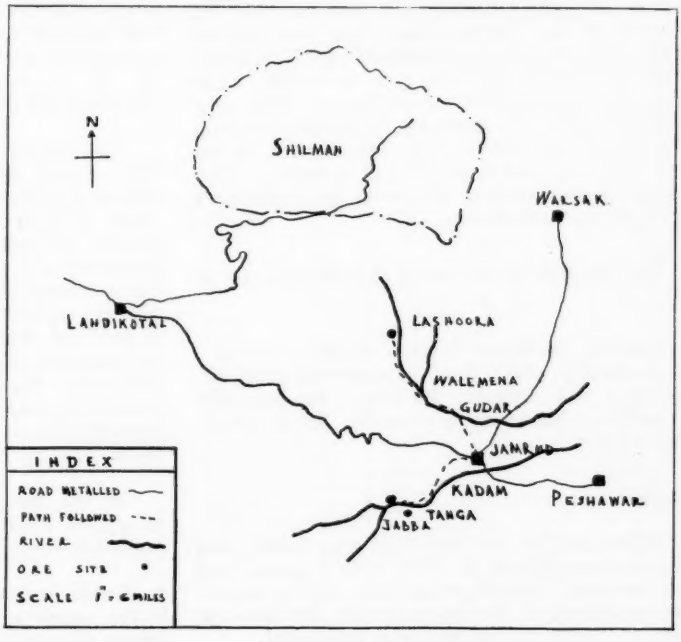

Fig. 1.—A part map of Khyber Agency.

measured was 10 to 12 ft. The physical properties of the mineral sample are as follows:

Colour: dark brown; streak: brown; lustre: metallic, dull; form: granular and compact; cleavage: not distinct; fracture: sub-conchoidal and uneven; hardness: 5.5 to 6; and specific gravity: 4.4.

B. Shilman

As the preliminary examination of the ore showed a poor percentage of iron mixed with a high percentage of alumina, silica, lime and magnesia, the detailed study of the ore has not been carried through.

Jabba

The topography is rugged and the dark massive limestone shows signs of weathering and denudation. Rocks are highly crumpled and shattered, mainly due to the action of heat and water. The massive limestone is shaly in nature and dark grey in colour.

Rusts and coatings of iron were found at several places, at some places appreciably thick but not of economic value. Huge rusts of iron were noticed at high altitude with several veins of hematite in it. The hematite vein near the top of the hill about 1800 ft. measured 30 ft. in length 15 ft. in thickness and 6 to 8 ft. in depth. The veins are interbedded with Eocene light grey limestone with the same dip as the mother rock. The second vein, which is in continuation of the first, is 5 ft. in thickness and 15 ft. in length. The whole rusting having the veins in it measured about 50 ft. in thickness.

The physical properties of the hematite are as follows:—

Colour: brownish black; streak: brown; lustre: metallic, dull; form: massive and compact; cleavage: not distinct; fracture: sub-concoidal to uneven; hardness: 5.5 to 6; and specific gravity: 3.3.

Tanga

When this site was visited, it was found that the only rust of iron in thick layer is spread out over the area of about 300 sq. yds. As the deposit was considered to be un-economic, the detailed study of Tanga ore has not been attempted.

2. Chemical Examination of the Ores

Samples collected from different places and even from the same area varied greatly in chemical composition, but some of the typical analyses are shown below:—

Table 1.—Chemical Analysis of the Ores.

Composition	Lashoora ore %	Shilman ore %	Jabba ore %
Loss on ignition	10.6	2.6	5.8
Silica	31.4	32.6	60.7
Iron oxide	51.4	23.9	31.1
Alumina	6.4	20.9	2.2
Lime	<0.1	9.2	—
Magnesia	<0.1	9.4	—

It will be evident from Table 1 that only the Lashoora ore is relatively rich in iron content. Since it also occurs in relatively abundant quantity, this ore was subjected to further detailed study particularly with reference to the very high percentage of objectionable silica. The results of this study are detailed below.

The Nature of Silica in the Lashoora Iron Ore.—With a view to examine the possibility of beneficiation, it was considered necessary to find out the nature of silica in the iron ore. First, the total silica in the sample was determined by the usual procedure of decomposing the finely ground sample in concentrated hydrochloric acid. The residue was filtered, washed and ignited. This gave the total of free and combined silica. In another sample, the separated silica was digested on the steam bath with 10% Na_2Co_3, filtered and washed. The residue obtained was free silica. The difference between free and total silica gave the combined silica.

In the sample under analysis, the total of 31.4% of total silica gave: Free silica, 22.9%; combined silica (by difference) 8.5%.

Ferrous Iron.—Ferrous iron was also determined in the sample to find out the possible existence of ferrous silicates by the following method.[1]

One gram of the finely powdered sample is decomposed in a current of carbon dioxide in a large platinum crucible with dilute sulphuric and hydrofluoric acids. When the decomposition is complete, the sample is taken in an excess of boric acid and promptly titrated with potassium permanganate (0.1 N).

Only a trace of ferrous iron is present in the iron ore.

3. Attempts at Beneficiating the Ore

The main objectionable impurity in the ore is the high percentage of silica (about 30%). If this could be reduced to the range of 10 to 12 percent, the ore would probably be easily reduced. The silica in the ore is partly combined (about 8.5%) and partly free silica (about 22.9%). While the combined portion cannot be separated by any physical process, efforts were made by some simple techniques to see whether the free silica could be separated or not.

Removal of Silica and Concentration of Ore by Simple Washing.[3]—Samples of 25 g. of one batch of powdered ore with 32.5% silica and 42.5% Fe_2O_3, passing through different B. S. sieves were washed successively 3 times with about 100 ml. of water each time and decanted after settling for about a minute. The results are given in Table 2.

TABLE 2

No. of experiment	Sample passing through B. S. mesh No.	% of recovered sample, dried at 105°C.	% of SiO_2 in the residue	% of Fe_2O_3 in the residue	% of reduction of silica	% of enrichment of Fe_2O_3
1	10	91.2	30.5	44.0	6.6	3.5
2	20	87.2	30.7	44.4	5.5	4.5
3	30	83.8	29.4	44.8	9.5	5.4
4	40	76.6	30.2	44.8	7.1	5.4
5	60	75.1	30.1	45.6	7.4	7.3
6	80	70.8	29.2	46.4	9.8	9.2
7	90	66.6	29.3	46.8	9.9	10.1
8	100	63.8	28.9	46.8	11.1	10.1
9	120	56.2	27.9	47.4	14.2	11.5
10	200	40.8	24.4	49.1	25.0	15.5

It is observed from the above that the recovery of the washed residue decreased with the fineness of the sample (*i.e.* higher mesh No.). The iron oxide content progressively increased and the silica percentage correspondingly decreased, but not to the desired limit.

A slightly different technique of washing was also tried by taking the powdered ore at the bottom of a long glass tube (120 cm. long and 5 cm. diameter) and overflowing the lighter particles by introducing water from the bottom (Fig. 2). The residue at the bottom did not show any better concentration of iron ore.

Gravity Separation.—As the specific gravity of hematite (5.2) is considerably higher than that of quartz silica (2.6), it was considered worth-while to try some kind of gravity separation. A liquid of specific gravity higher than the specific gravity of silica, would have been ideal, such as tetrabromoethene, methyl iodide etc.; but as these were not available, a concentrated solution of calcium chloride was prepared of sp. gr. 1.8. The powdered ore was shaken with this solution in a cylinder and allowed to settle. After partial settling, the top portion of liquid was decanted and the residue was washed free from calcium chloride and analysed. The concentration effect of this process was not found to be significantly higher than that of the simple washing method.

A horse-shoe magnet also did not attract

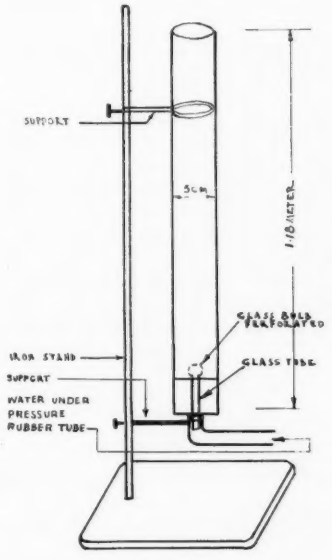

Fig. 2.—Gravity ore washer.

preferentially the iron oxide particles from the powdered ore.

Study under the Microscope.—The powdered sample of the ore under the microscope showed clearly the crystals of hematite and sand particles, although there were some large segregated particles of hematite, the majority of them in small particles were found intimately mixed with the sand particles. This fine admixture makes the physical separation of hematite and silica so difficult.

4. Conclusion and Lines for Further Study

The observations carried out by us indicate strongly against the possibility of reduction of iron ore found in Khyber Agency and the difficulty of beneficiation by the usual method of ore dressing. Further geological study is needed to find out if better quality iron ore is available at a greater depth from the surface. Samples collected and investigated by the authors were mostly from the surface.

It is also worth-while to study the possibility and economics of producing ferrosilicon from the ore—which appears suitable for the production of the alloy; a furnace to produce ferrosilicon requiring a much higher temperature than that required at the blast furnace—is at present not available in these Laboratories.

Acknowledgements.—The authors desire to acknowledge with thanks the encouragements and suggestions by Col. M. K. Afridi and also Prof. Mirza Anwar Beg for his collection of the samples and erection of a cupola furnace.

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A STUDY OF MULTANI MITTI *

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No work has yet been done for the identification of the Khairpur clay, better known as Multani mitti, which is believed to be fuller's earth. The author has investigated the mineralogical composition and tried to estimate the different components in order to facilitate scientific utilisation. The investigations show that Multani Mitti agrees well with Attapulgite Palygorskite, a clay mineral found in fuller's earth from Attapalgus, Georgia etc. The relative percentages of different components have not been determined precisely, but free silica was found to be 2.75% (± 0.25%).

Introduction

There is a vast deposit of clay in Khairpur, which is known as "Multani mitti". This clay is widely used for different purposes, and is generally believed to be fuller's earth. No work has yet been done for its identification, and it was therefore thought worthwhile to study the mineralogical composition and if possible to estimate the different components in order to facilitate its scientific utilization.

Information on the geology of the area is meagre, but from the scanty literature available it may be said that the land is very young geologically, belonging to the tertiary and post-tertiary periods. It is formed by the upheaval of the land from Tethys Sea, which once covered the whole of Western Pakistan, but gradually withdrew with the rising of the Himalayas and its flanks.[1] The sedimentary nature of the clay can be easily recognized by its stratification.

1. Chemical Composition

The chemical composition of the representative specimen is as follows:

SiO_2, 51.50%; Al_2O_3, 19.06%; Fe_2O_3, 7.31%; TiO_2, 1.19%; CaO, 0.39%; MgO, 2.51%; K_2O, 2.60%; Na_2O, 0.99%; loss on ignition, 8.90%.

The silica-alumina ratio is about 3.

2. Identification By X-ray Diffraction

The reference specimen is yellow in colour, apparently amorphous powder. Eighty-four percent of the clay is less than 4 microns, and the pretreatment, using water for separation of clay fraction, was not effective due to very small particle size. It was packed in plastic specimen holder and analysed by X-ray diffractometer (Seifert). The instrumental setting used was as follows:

Counting rate, 3000 counts/second; time constant, 3 seconds; Geiger over-voltage, 1500 volts; divergence slit, 1.5; receiving slit, 4.5; scatter slit, 0.5; scanning speed, 5′/1°; chart scale, 300 mm./h.; radiation Cu. Kα; (15 mA-40 kV).

The principal reflections can be indexed as follows:

10.495 A°	4.49 A°	3.24 A°	2.57 A°
(110)	(040)	(400)	(510)

which corresponds to attapulgite. The diffraction diagram is given in Fig. 1. Quartz, kaolin and limonite are fine grained impurities. Leaching with dilute hydrochloric acid was not effective for the removal of limonite. Digestion of the clay with concentrated hydrochloric acid for 4 hours on water bath removed most of the limonite giving a pale cream product. The structure of attapulgite was resistant to the acid treatment and gave the same reflections except that the quartz reflections were sharper.

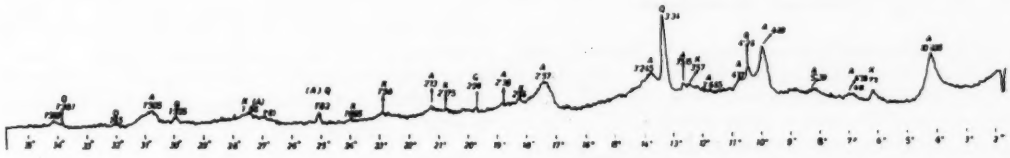

Fig. 1.—X-ray diffraction diagram of Multani mitti.

*The major part of the work described in this communication was carried out at the laboratories of Societe Francaise de Ceramique during the tenure of a French Government Fellowship in the year 1959.

(*i*) *Effects of Treatment with Ethylene Glycol.*—This caused the 10.495 A° reflection to be replaced by one at 10.625 A°. The general nature of other reflections remained the same. The procedure is as follows:the pulverized specimen was treated with drops of ethylene glycol, mixed thoroughly with a glass rod, packed on a plastic holder, then studied by X-ray.

(*ii*) *Effects of Treatment with* NH_4NO_3.—Powdered specimen was boiled in N ammonium nitrate solution for 10 minutes and studied by X-ray. No change was observed.

(*iii*) *Estimation of* SiO_2 *(Free)*.—Estimation of free silica by chemical methods gives disappointing results. The method of Legrand and Nicolas[2] was followed to estimate the quartz content of Multani mitti.

The height of the peak of 25° reflection of quartz was measured (using Al 41° as external standard). This height gave the percentage of quartz from the standard curve. The standard curve was obtained by plotting the relative heights of quartz reflections against the percentage of quartz mixed with illite (attapulgite is more related to illite than any other clay mineral). This method gave the percentage of quartz in Multani mitti as 2.75% (± 0.25%).

3. Electron Micrographic Examination

The elctron microscope has permitted the precise determination of the shape of various clay minerals. A thin membrane of parlodion (2% in amyl acetate) was coated on a grate. Dried by passing infra-red rays. Two mg. of the clay was shaked with 5 ml. of distilled water. One drop of this clay suspension was taken on the grate and dried in the same manner. It was studied under the electronic microscope.

The electronic micrograph of Multani mitti shows single laths and bundle of laths (Fig. 2). There is no evidence of tubular form (like that of halloysite). Small, poorly defined flakes, grouped together in irregular aggregates may be due to the presence of illite. Big hexagonal crystals seem to be that of kaolinite (or may be of quartz).

4. Thermal Treatment

(*i*) *Differential Thermal Analysis.*—Thermal analysis shows the following characteristics. (Fig. 3).

(*a*) Endothermic reaction between 20°C. and 250°C., with a peak at 150°C.

(*b*) Endothermic reaction between 425°C. and 700°C., with a peak at 575°C.

Fig. 2

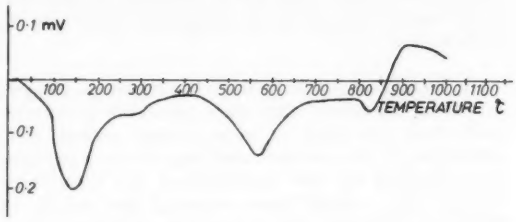

Fig. 3.—Differential thermal analysis curve. Rise of temperature: 400°C./hr.

(*c*) Endothermic reaction between 800°C. and 865°C., with a peak at 830°C.

(*d*) Exothermic reaction starts at 865°C. and peak is attained at 910°C.

When HCl-treated clay was analysed thermally, the general nature of the curve remained the same except slight changes in temperature ranges of endothermic and exothermic peaks. The area of the first endothermic inflection has decreased in comparison to that of the untreated one.

The sample was reheated to study its behaviour. It gave a peculiar curve (Fig. 4).

(*ii*) *Dehydration Curve.*—The weight loss curve obtained by continuous recording (Fig. 5) shows a heavy loss between 20°C. and 250°C. which corresponds to the first endothermic peak. Then it slows down to 325°C. Between 325° and 600°C. again there is a heavy loss. After 600°C. the loss is almost negligible.

(*iii*) *Dilatation Curve.*—The dilatation curve

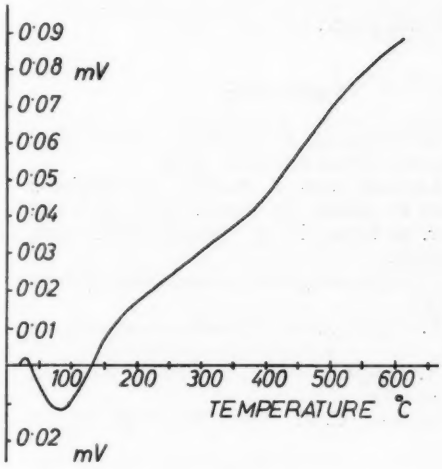

Fig. 4.—Differential thermal analysis curve. Rise of temperature: 4000° C./hr.

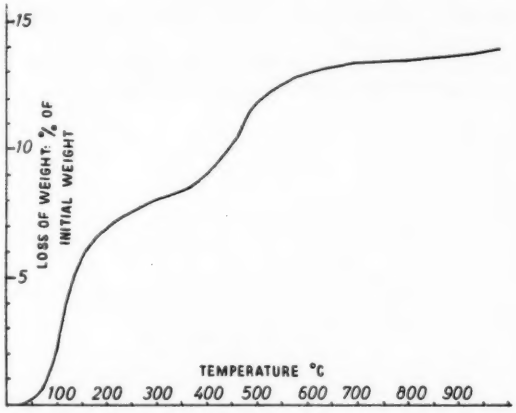

Fig. 5.—Dehydration curve.

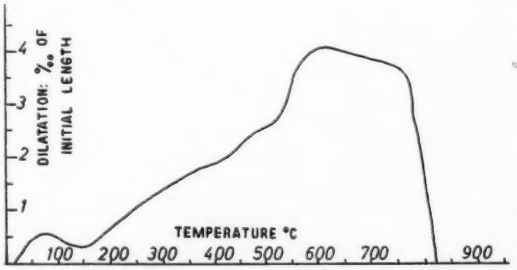

Fig. 6.—Dilatation curve.

shows the following facts (Fig. 6):

(*a*) Slight contraction between 60°C. and 150°C.
(*b*) Slight contraction between 400° C. and 450°C.
(*c*) Slight contraction between 525°C. and 550°C.
(*d*) Maximum expansion (4.25‰ of the initial length) was achieved at 615°C., and then slight contraction continued till 775°C.
(*e*) Large contraction after 775°C.

4. Discussion

In the light of the above obtained X-ray data Multani mitti agrees well with attapulgite-palygorskite first described by De Lapperant.[3] He

Table 1.—Powder Data for Attapulgite (Palygorskite).

	Data by Bradley (1940)							
	Data calculted from proposed structure		Experimental data		Experimental data by Lapparent		Multani mitti	
hkl	d	I	d	I	d	I	d	I
110	10.48	330	10.50	10	10.2	12	10.495	70
200	6.45	17	6.44	6	6.44	2	6.45	6
130	5.44	12	5.42	5	5.3	2	5.44	10
220	5.24	1						
040	4.50	66	4.49	8			4.49	100
310	4.18	13	4.18	3	4.3	10	4.17	25
240	3.69	18	3.69	5				
330	3.49	12	3.50	3			3.52	15
150	3.47	2						
400	3.23	120	3.23	10	3.25	10	3.25	40
420	3.04	3	3.03	1				
350	2.76	2						
440	2.62	43	2.61	8				
510	2.56	8	2.55	3	2.55	10	2.58	60
530	2.38	5	2.38	3			2.38	13
080	2.25	1						
600	2.15	15	2.15	5	2.25	4	2.13	13
550	2.10	1						
480	1.845	1						
390	1.815	3	1.82	1	1.80	2	1.82[q]	13
650	1.75	1						
800	1.615	3	1.62	1	1.67	2	1.66[k]	17
680	1.555	17	1.56	3				
0120	1.50	40	1.50	5	1.49	4	1.50	30

d: spacing I: intensity, q: attapulgite masked by quartz, k: attapulgite masked by kaolin.

found this clay mineral in fuller's earth from Attapulgus, Georgia, Quincy, Florida and Mormoiron (France). Bradley[4] has determined the structure of the mineral, showing silica chain similar to those in amphibole to be essential components of its structure. Bradley[4] gives the ideal formula of attapulgit[4] as $(OH_2)_4$ $(OH)_2$-Mg_5 Si_8 O_{20}. $4H_2O$ in which there is a considerable replacement of Mg by Al.

The X-ray data obtained by the author, by De Lapparent[5] and by Bradley are collected and compared in Table 1.

The electron micrograph clearly confirms the attapulgite nature of clay. But illite as an impurity cannot be ruled out. Nothing can be said about the relative percentage of different component except that of free silica which was found to be 2.75% (± 0.25%).

The general shape of the curves obtained by differential thermal analysis, dehydration analysis and dilatation also agrees with those of attapulgite obtained by Caillere,[6] Caillere and Henin,[7] De Lapparent,[3] Longchambon[8,9] and Alexanian.[10]

Acknowledgements.—The author is grateful to Mr. Paul Paquin, Directeur Generale, Societe Francsaise de Ceramique, Paris for his permission to publish this paper.

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AN INVESTIGATION OF THE INFLUENCE OF HUMIDITY AND WATER CONTENT ON THE ELECTRICAL PROPERTIES OF COTTONSEED CAKE

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In the course of investigations on the development of a rapid non-destructive electrical method for the estimation of oil content of a single cottonseed, the dielectric constant of cottonseed cake was found to rise about 50-fold when the dry cake absorbed water up to its saturation value 12-16%. The present communication deals with a detailed investigation of the nature of this variation of the dielectric constant and also the specific conductance of variously formed discs of cottonseed cake, using several different relative humidities.

The measured values of both the dielectric constant K and the specific conductance γ under various humid conditions are found to increase exponentially with the percentage water constant W of the cake and can be represented by the following equations $K = 4 \exp (26 \times w/100)$, $\gamma = 0.2 \exp. (56 \times w/100)$. Further work is in hand for the elucidation of this type of variation, particularly with reference to other hydrated solids.

In the course of earlier investigations [1,2] relating to the development of a rapid non-destructive electrical method for estimation of the oil content of a single cottonseed, it was found that the dielectric constant 'K' of a typical cottonseed cake rises about fifty-fold from nearly 3 to about 140 as the dry cake takes up water up to its saturation value of 12-16%. Figure 1 shows the observed variation of 1/K plotted against the relative humidity and the percentage water content, and it was considered of interest to follow up this finding with a somewhat fuller investigation of the nature of the variation of both specific conductance and dielectric constant in humidified cottonseed cakes, which may be looked upon as a simple protein-water system.

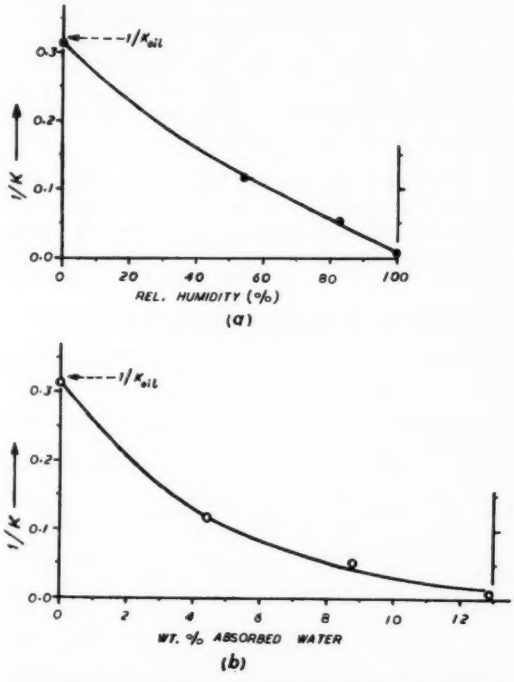

Fig. 1.—Previously noted variation of 1/K, the reciprocal of the dielectric constant of cottonseed cake, against (a) relative humidity of the atmosphere, and (b) percent water absorbed by the cake.

1. Experimental Procedure

For this work, two types of samples were prepared in the shape of 6 cm. diameter discs with a thickness of 5 to 7 mm. Firstly, the sample used in the previous experiments, which was made from commercially available cottonseed cake after washing with petrol, and sieving to free it from bits of the hulls. The clean cake had then been compacted in a die at 100°C. under light pressure in a laboratory hydraulic press. The disc of cake obtained was found to have a density of 0.90 g./ml. The second sample disc was prepared from dehulled kernels of the M 4 variety of cottonseed after Soxhlet extraction of the oil. This method yielded a more compact disc, of density about 1.1 g./ml. For the dielectric constant and conductance measurements, these discs were placed between the two plates (2.5 cm. square) of the parallel-plate condenser used in the previous investigations, and connected by short stiff copper leads to the measuring terminals of the Marconi universal bridge. The plates of the condenser were adjusted to just touch the two faces of the sample disc, the criterion being that the disc should stay in place without external support but be capable of being withdrawn by a light touch.

The capacity 'C' of the condenser was measured on the bridge at 1,000 cycles per second, both with and without the sample disc, and the dielectric constant could be calculated from the ratio of

these two values. The values of the phase angle δ were also measured with the bridge, and from these the conductance of the sample could be calculated by means of the formula.

$$w\,C \tan \delta = 1/R \qquad (1)$$

where $1/R$ = the conductance and $w = 2\pi f$, f being the frequency, in this case 1000 cycles per second.

A simple but significant correction is required for the stray capacity due to the leads and strips making connection with the actual condenser plates. This stray capacity, Δ, is to be subtracted from all the above values read on the bridge, and was determined experimentally by measuring the capacity of the (air) condenser for several values of the distance 'd' between the plates. If the area of the plates is 'A' , then the measured value C_{air} will be

$$C_{air} = \frac{A}{4\pi d} + \Delta \qquad (2)$$

So that the graph for C_{air} against $1/d$ should be a straight line extrapolating to the value Δ for $1/d = 0$. Such a graph is shown in Fig. 2 and yields the figure of $0.4\mu\mu F$ for Δ, which is to be compared with an estimate of 0.3, calculated from the configurations of the leads, etc. (cf. also the measured capacity of about $2\mu\mu F$ for the condenser when d = 0.5 cm., *i.e.* 5 mm.)

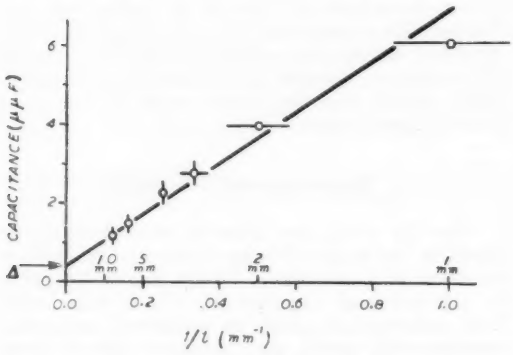

Fig. 2.—Graph for capacitance of measuring condenser against $1/t$, extrapolated to yield a measure of the stray capacitance, Δ. The short vertical and horizontal lines through the points represent the observational errors.

2. Results

Starting with the sample discs at atmospheric humidty (80%) the capacitance and conductance were measured as described above, and the dielectric constant K and specific conductance γ calculated. Each sample was then put through a complete hydration-dehydration cycle by placing it in various constant humidity chambers, and the electrical constants were measured after 20 to 30 hours when equilibrium had been very nearly attained with the atmosphere of the appropriate chamber. Each measurement was repeated two or three times. The weight of absorbed water was obtained by weighing the samples at each stage and subtracting the weight of the completely dried sample disc; the % water was calculated on the basis of the total weight.

The results for the two samples are shown in Tables 1 and 2, together with the experimentally estimated standard deivations. Since the total variation in K and γ is several hundred-fold, the last two rows of each table give the calculated

TABLE 1.—MEASURED ELECTRICAL CONSTANTS OF SAMPLE NO. 1 AT DIFFERENT WATER CONTENTS.

	1	2	3	4	5	6	7	8
Relative humidity (%)	80	100	30	0	85	85	—	0
Weight of water (g)	1.21	2.73	0.32	0.00	0.76	0.78	0.19	0.06
% of water= w	7.7	15.8	2.2	0.0	5.0	5.1	1.3	0.4
K	19±2	125 ±10	4.3±0.2	3.4±0.0	24±2	28.5±2	3.5±0.2	2.8±0.1
γ (mμ Mho/cm.)	10.5±1	1050±100	0.8±0.1	0.2±0.1	14.2±1	17.0±1	0.5±0.1	0.3±0.04
ln K	2.94±0.1	4.83±0.1	1.46±0.05	1.22±0.06	3.18±0.08	3.35±0.07	1.25±0.06	1.03±0.04
ln γ	2.35	6.97	$\bar{1}$.78	$\bar{2}$.39	2.65	2.83	$\bar{1}$.22	$\bar{2}$.30

values of the Napierian logarithms of the dielectric constant 'K' and the conductance γ.

Firstly, it is seen that the large change in K is accompanied by an equally large change in the conductance. The changes in the electrical quantities are similar for the two samples and are on the whole reversible when the samples are taken through a complete hydration-dehydration cycle. This is clearly brought out by the graphs of Fig. 3, in which the logarithms of K and γ are plotted against weight % absorbed water, the triangles and circles corresponding to the two specimens of Tables 1 and 2, respectively, while the subscript numbers correspond to the column nos. in these tables. It is seen that, with rising or falling water-content, there is no definite bias for the plotted points to lie on any one side of the mean graph, and therefore no indication of irreversible changes.

3. Discussion

Taking into account the tabulated standard deviations of ln K and ln γ, which are indicated by the short vertical lines in Fig. 3, the straight lines shown can be drawn and the following equations derived to fit the experimental data:—

$$\ln K = 1.18 + 26 \times w/100$$

i.e.

$$K = 4 \exp.\left(26 \times \frac{w}{100}\right) \qquad (3)$$

and

$$\ln \gamma = -1.8 + 56 \times w/100$$

i.e.

$$\gamma = 0.2 \exp.\left(56 \times \frac{w}{100}\right) \qquad (4)$$

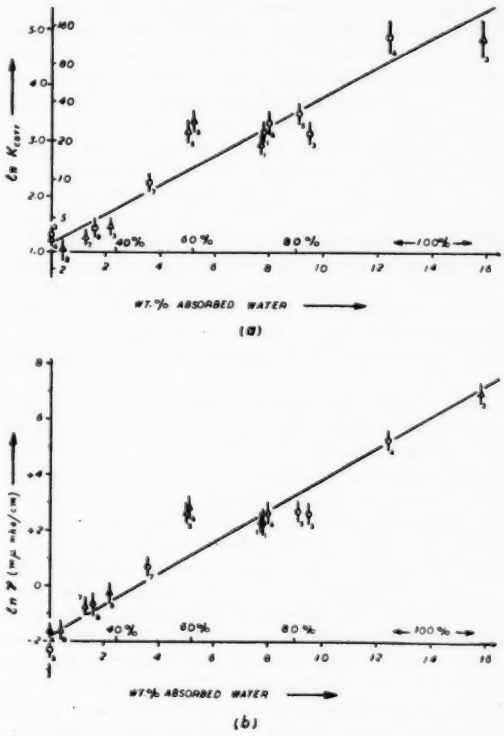

Fig. 3.—Semi-logarithmic plots of (a) the dielectric constant, K, and (b) the specific coductance, γ, of the two samples of cottonseed cake, carried through about one and a half hydration-dehydration cycle. The triangles are for the sample of Table 1, and the circles for the sample of Table 2; the short vertical lines through the plotted points indicate the estimated standard deviations, while the subscripts to them correspond to the numbers heading the columns in Tables 1 and 2.

TABLE 2.—MEASURED ELECTRICAL CONSTANTS OF SAMPLE NO. 2 AT DIFFERENT WATER CONTENTS.

	1	2	3	4	5	6	7	8
Relative humidity (%)	80	0	85	100	85	85	—	0
Weight of water (g)	1.01	0.00	1.24	1.69	1.20	1.03	0.44	0.19
% of water= w	7.8	0.0	9.5	12.5	9.1	8.0	3.6	1.6
K	24±2	3.7±0.2	23±2	130±15	33±4	28±3	9.6±0.5	4.2±0.2
γ (m μ Mho/cm.)	10.1 ±1	0.1	14.0 ±1	198±15	15±1	13.7±1	2.0±0.02	0.5±0.1
ln K	3.18±0.08	1.31±0.06	3.14±0.08	4.87±0.12	3.50±0.12	3.33±0.10	2.26±0.05	1.44±0.05
ln γ	2.30	3.70	2.64	5.29	2.71	2.62	0.69	1.37

These equations show that both the conductivity and the dielectric constant vary exponentially with the percentage water content w of the cake, and further that the conductivity varies nearly as the square of the dielectric constant, because the coefficient of w in equation (4) is nearly twice that in equation (3). This latter point is further confirmed by the plot of Fig. 4 for ln γ against ln K, whose slope is 2.02 ± 0.15. A noteworthy feature of Fig. 4 is that the scatter of the experimental points about the straight line is considerably less than for Fig. 3, in which there is probably a component of error due to lack of complete equilibrium of the absorbed water. This is supported by Fig. 5, showing the data of Tables 1 and 2 plotted as 1/K against relative humidity.

Since both the samples used in the foregoing experiments were subjected to treatment with petrol and heating to over 100°C. under the applied pressure, the presence of living cells in the pressed cake can be discounted, and therefore the very large variations in K and γ are to be interpreted in terms of the elctrolytic action of the absorbed water. The effect can be expected to increase non-linearly with the quantity of the water because the degree of dissociation and ionization of the proteinous molecules will also increase rapidly from zero as the water is added. While the full mechanism of this is yet to be worked out, considerable support for these ideas is obtained form the fact that an approximately exponential dependence of conductivity on percentage water is observed in the case of very highly concentrated aqueous solutions of salts at concentrations of 15 to 20 equivalent weights per litre of solution.[3] Figure 6 depicts this data[3] for zinc chloride on a semi-logarithmic graph, and the tendency of the graphs for specific conduc-

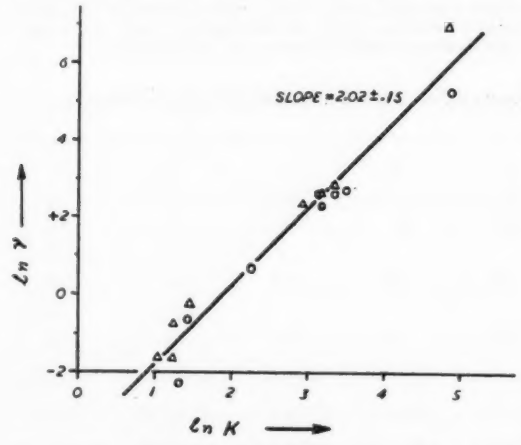

Fig. 4.—Plot showing the linear relationship between ln γ and ln K, the slope being almost exactly 2; the circles and triangles correspond as before to the two samples.

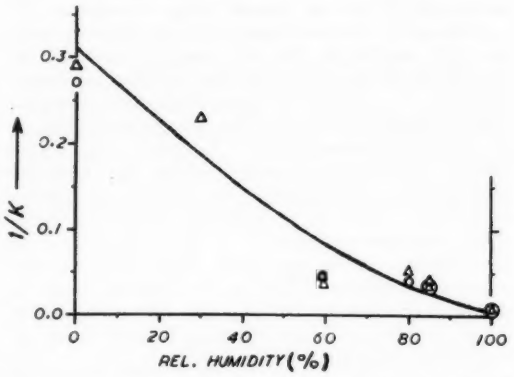

Fig. 5.—Plot of 1/K against percent relative humidity, based on the present data. Except for the scatter of the points, the graph is in good agreement with that of Fig. 1 (a).

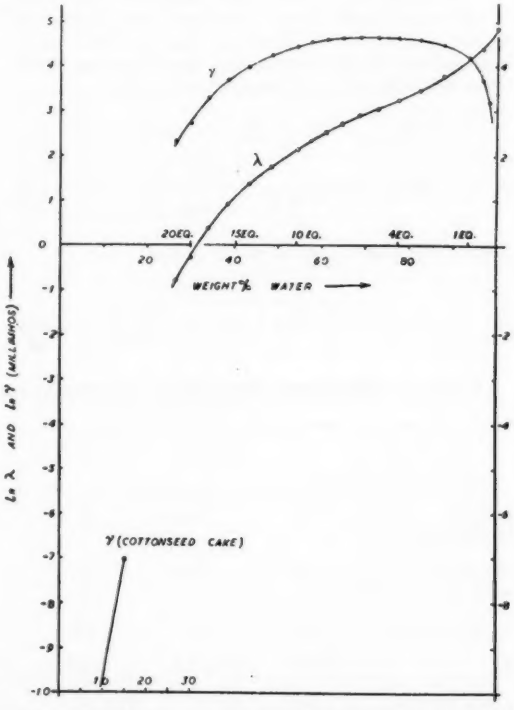

Fig. 6.—Standard data on conductance of concentrated solutions of zinc chloride, plotted semi-logarithmically for comparison with the results obtained with cottonseed cake in the present paper (bottom of figure). The possibility of a general connection between the two is strongly suggested by the graphs.

tance γ as well as equivalent conductance λ towards inclined straight lines at water contents of less than 35% is clearly marked. Moreover, the slopes estimated at 15% water content are of the same order of magnitude as in the case of the graph of Fig. 3 for cottonseed cake.

These results indicate the desirability of a thorough investigation of the influence of small percentages of water on the electrical properties of various solid compounds before a full interpretation of the electrical conduction mechanism is put forward.

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CHEMICAL STUDIES IN RELATION TO THE BIOSYNTHESIS OF SOME NATURAL BENZOFURANS AND RELATED COMPOUNDS. PART II*

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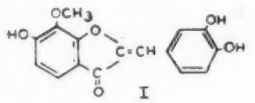
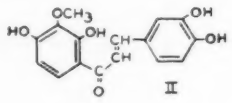
(Received December 2, 1959)

4′-Methoxybenzylidene-coumaran-3-one (VI) on treatement with alcoholic alkali gave anisaldehyde. The other products obtained were the flavone (VIII) and a diketone (IX). Reduction of (VI) gave 2(4′-methoxy-benzyl)-3-hydroxy-dihydrobenzofuran. These reactions provide some chemical basis for the hypothesis that benzalcoumaranones probably derived by the condensations of benzaldehydes and coumaranones are involved in the biosynthesis of flavones and related substances.

Introduction

Little work of a biochemical nature has been done on the possibility of biosynthesis of flavones and related substances including furanoid compounds from benzalcoumaranones, but it appears certain that oxidation processes play an important role in the formation of these compounds in plants.[1-6] The present investigation while primarily concerned with providing some further chemical evidence in support of the hypothesis that coumarones could be derived from benzalcoumaranones has also produced some unexpected and at the same time quite interesting results. One of the results which has been of particular significance was the formation of the flavone (VIII) from benzylidenecoumaranone (VI) by the action of alcoholic alkali. Leptosidin (I), a benzalcoumaranone, has been isolated from *Coreopsis grandiflora* and it is significant that 2′, 4′, 3, 4-tetrahydroxy-3′-methoxy chalcone (II) has also been isolated from this plant. The co-occurrence of the coumaran-3-one aureusidin (III) and the flavone luteolin (IV) in *Antirrhinum majus* is equally noteworthy.


I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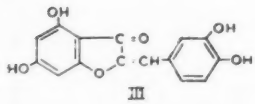
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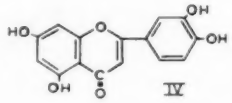
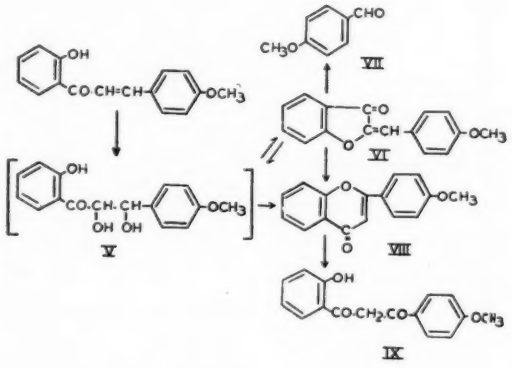

IV

Such co-existence and structural relations, besides indicating the biosynthetic relationships among the different groups of compounds mentioned above, also suggest that coumaran-3-ones may be derived from benzylidenecoumaranone by loss of the benzylidene group. The most plausible mechanism for the removal of the benzylidene group would be, however, by means of a reverse-aldol reaction as shown in the case of 6 - methoxy - 2 - isopropylidene-coumaran - 3-one Reduction of coumaran-3-one with sodium borohydride could then give coumarone (cf. Part I of this series).

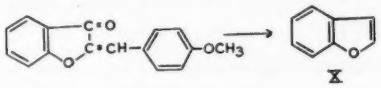
The oxidation process mentioned above appears to be vitally connected with the formation of flavones and related substances from benzalcoumaranones as shown in the following scheme.



Although the co-occurrence of benzalcoumaranone and flavone is less frequently encountered in nature, the one in *Autirrhinum majus* indicates that these compounds are interconvertible. The most probable mechanism involved in this interconversion would be, however, dependent upon the opening of the ring and formation of the intermediate (V). Formation of the flavone from benzalcoumaranone in the laboratory lends support to this view. While it may be argued that the reaction is not analogous to a cellular process it is certain that the same results could be achieved under conditions approximating to those obtaining in living cells.

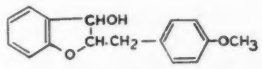
*The work described in this paper was carried out in the Department of Organic Chemistry, Univeristy of Sydney, Australia.

Reactions of 4′ - Methoxybenzylidene - coumaran - 3-one.—In order to provide some basis for a hypothesis such as that above, the reactions of 4′ - methoxy - benzylidene - coumaran - 3 -one (VI) were studied. Reduction of (VI) with sodium borohydride[7] was expected to yield coumarone (X) and/or related substances.



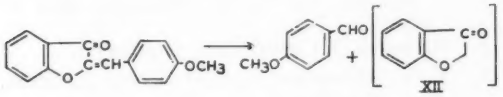
X

The only product of this reaction was a substance $C_{16}H_{16}O_3$ to which structure (XI) is assigned. An attempt to dehydrate it with dilute sulphuric acid proved unsuccessful.



XI

4′ -Methoxybenzylidene - coumaran - 3 -one (VI) was then examined to see whether a reverse aldol reaction could remove the benzylidene group as anisaldehyde (VII) and leave coumaran-3-one (XII) or a derivative.



XII

Treatment with very dilute alkali resulted only in the recovery of the starting material. Prolonged treatment with alcoholic alkali at room temperature gave mainly anisaldehyde which was indentified as its 2:4-dinitrophenylhydrazone. It seems likely that coumaran-3-one formed under went degradation under the influence of alkali leading to the production of an unidentifiable polymeric material. A side reaction also occurred resulting in the formation of the flavone (VIII) and a diketone, 2-hydroxybenzoylanisoyl methane (IX), identified by mixed melting point with an authentic specimen prepared by a standard method[8].

Experimental

Melting points are uncorrected. Microanalyses were carried out partly by the C.S.I.R.O. Microanalytical Laboratory and partly by the Microanalytical Laboratory, University of Sydney.

(*a*) *4-Methoxy -2′ -hydroxychalcone.*—*o*- Hydroxyacetophenone (13.6 g.) was added to an aqueous alcoholic solution of sodium hydroxide (10 g.; water, 80 ml; ethanol, 50 ml.). The solution was stirred and anisaldehyde (13.6 g.) slowly added, the temperature being maintained at 25°C. Vigorous stirring was continued for three hours at this temperature , and then the mixture was kept at 0°C. overnight. The solid yellow mass was acidified with acetic acid (15%). 4-Methoxy - 2′- hydroxychalcone, m.p. 93-4°C., from methanol was obtained in good yield.

(*b*) *4 - Methoxy - 2′ - hydroxychalcone Dibromide.*—4 - Methoxy - 2′ - hydroxychalcone (10 g.) was dissolved in a minimum amount of carbon disulphide and the solution was cooled to 0°C. Bromine (20 ml.; 1 vol. bromine; 9 vol. carbon disulphide) was added with stirring, the temperature being kept below 10°C. The dibromide separated as granular yellow crystals, m.p. 137-8°C., from methanol.

(*c*) *4′ - Methoxybenzylidene - coumaran - 3 - one.*—A mixture of the dibromide (10.5 g.) and absolute ethanol (200 ml.) was heated to boiling on a water bath. When the solid was completely dissolved sodium hydroxide (50% ; 10 ml.) was added and the mixture refluxed for ten minutes. The solution was immediately cooled and diluted with water. The crude product was filtered and washed severeal times on the filter with water. 4′-Methoxybenzylidene-coumaran-3-one, m. p. 138-9°C. from methanol was obtained in 60% of the theoretical yield.

(*d*) *Reduction of 4′ - Methoxybenzylidene coumaran - 3 - one with Sodium Borohydride.*—4′ - Methoxybenzylidene - coumaran - 3 - one (500 mg.) was dissolved in absolute ethanol (25 ml.) by warming on a water bath. The system was filled with nitrogen and sodium borohydride (300 mg.) in ethanol added. The mixture was refluxed one hour. After cooling it was acidified with dilute acetic acid, diluted with water and extracted with ether. The combined extracts were washed with sodium bicarbonate solution and finally with water. Evaporation of the solvent gave an oily residue which cyrstallised on standing.

2(4′ - Methoxybenzyl) - coumaran - 3 - ol, m.p. 114-5°C., was recrystallised from light petroleum (b.p. 60-90°C.). (Found: C, 74.7; H, 6.1. $C_{16}H_{16}O_3$ requires C, 75.0; H, 6.3).

(*e*) *Alkaline Treatment of 4′ - Methoxybenzylidene-coumaran - 3 - one.*—4′ - Methoxybenzylidene - coumaran - 3 - one (1 g.) was dissolved in ethanol (50 ml.) and sodium hydroxide solution (50 ml.; 20%) added. The mixture was allowed to stand twenty-four hours at room temperature. After dilution with water it was extracted with ether.

Evaporation of the solvent gave a mixture of solid and oil which was diluted with a little methanol and filtered. 4′ - Methoxy - flavone, m.p. 155°C., was recrystallised from methanol. (Found: C, 76.2; H, 4.9. $C_{16}H_{12}O_3$ requires C, 76.2; H, 4.8).

The methanolic mother liquors containing the oil gave a derivative with 2:4-dinitrophenylhydrazine hydrochloride, m.p. 250°C., not depressed in mixed melting point with an authentic specimen of anisaldehyde 2:4-dinitrophenylhydrazone.

The alkaline solution was acidified and extracted with ether. After evaporation of the solvent, *o*-hydroxybenzoyl-anisoyl methane, m.p. 111°C., from methanol, not depressed in mixed melting point with an authentic specimen was obtained. (Found: C, 70.7; H, 5.4. $C_{16}H_{14}O_4$ requires C, 71.0; H, 5.2).

(*f*) *o - Anisoyloxy - acetophenone.*—*o*- Hydroxyacetophenone (5 g.) was dissolved in pyridine (10 cc.) and anisoyl chloride (6.5 g.) added. The mixture was heated fifteen minutes on a water bath and allowed to stand at room temperature for one hour. It was then poured into dilute hydrochloric acid (2N). *o* - Anisoyloxyacetophenone separated in good yield (8 g.), and was recrystallised from ethanol, m.p. 111°C.

(*g*) *o- Hydroxybenzoyl - anisoyl - methane.* —*o* - Anisoyloxy - acetophenone (5 g.) was added to powdered sodium (400 mg.) in dry toluene (35 ml) and refluxed three hours. After cooling the mixture was acidified with dilute acetic acid (1:1). The aqueous layer was separated and the toluene diluted with ether and extracted with sodium hydroxide (5%). Acidification of the alkaline extract resulted in the separation of *o* - hydroxybenzoyl - anisoyl - methane, m.p. 112°C., from methanol.

Acknowledgements.—The author wishes to express his gratitude to Professor A. J. Birch for his kind help and advice and also to Dr. E. Ritchie and Dr. P. Elliott for many helpful suggestions. Finally he would like to thank the Colombo Plan Authorities for a fellowship.

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ATTEMPTED SYNTHESIS OF EVODONE*

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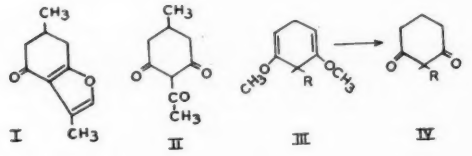
α-Bromoacetal and α-bromopropionacetal were condensed with 1,3-dimethoxy-2,5-dihydrobenzene, and its toluene homologue and the resulting products cyclised.

The product obtained by condensation of dihydroorcinol dimethyl ether with α-bromopropionacetal on cyclisation gave evodone possibly mixed with the cyclic half acetal.

Introduction

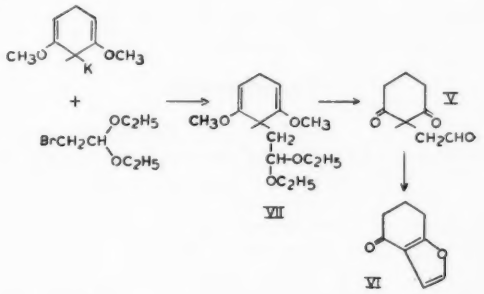
The structure of evodone (I)[1] was based on degradative evidence obtained by van Hulsen[2] (1941) which included ozonolysis to the crystalline compound (II), the structure of which was suggested and the synthesis carried out by Birch and Rickards.[1] The ultra-violet spectrum of evodone was also available. It was not possible, however, to obtain authentic specimens of evodone or its degradation products for comparison purposes and to that extent the structure of evodone is still uncertain. To provide further evidence it was felt that the synthesis of (±) evodone (the natural material is optically active) would enable, in particular, the ultra-violet spectrum to be examined, and the rather unusual ozonolysis to (II) to be repeated.



I II III IV

The synthesis has been based on the reaction discovered by Birch[3] that 2:5 - dihydro - 1:3 - dimethoxybenzene (III) (R = H) forms a potassium salt (III) (R = K) with potassium amide in ammonia, which in turn can be alkylated by an alkyl halide RX to (III) (R = alkyl). Being an enol-ether, on acid hydrolysis this produces (IV) (R = alkyl).

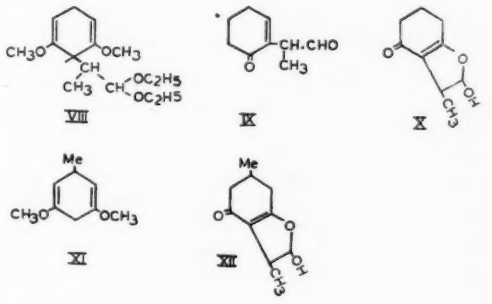
In the present case the type of intermediate desired is (V) which would be expected to ring-close to (VI). The sequence of reactions shown in the diagram was, therefore, examined in the first instance as a model. Alkylation to (VII) appeared to proceed normally, but difficulty was experienced in obtaining (VI). The



VII V VI

product of the reaction analysed rather badly for a content of three oxygens. It did, however, give rise to a semicarbazone, m.p. 195-196°C., and a 2:4 - dinitrophenyl-hydrazone, m.p. 247-248°C., analysing as derivatives of (VI). The crude oil also showed the ultra-violet maximum ($\lambda_{max.}$ 266 mμ, log. ε max. 3·84) to be expected of (VI) on the basis of the absorption of evodone ($\lambda_{max.}$ 265 mμ; log. $\varepsilon_{max.}$ 3·57).

Condensation of (III) (R = K) with α-bromopropionacetal produced the expected (VIII) which on mild acid treatment gave a product $C_9H_{12}O_3$ which may be (IX) or (X).



VIII IX X

XI XII

The action of 2N hydrochloric acid caused some loss of oxygen, the product analysing between $C_9H_{10}O_2$ and $C_9H_{12}O_3$.

*This work was carried out in the School of Chemistry, University of Sydney, Australia.

It was further found that the orcinol derivative (XI) condensed with bromoacetal in the expected manner, and the product of treatment with 2N hydrochloric acid had the expected ultra-violet spectrum ($\lambda_{max.}$ 269 mμ; log. ϵ max. 4.433). The dihydro-orcinol dimethyl ether (XI) with α-bromopropionacetal produced a substance probably containing some of the expected compound, but analysing badly. Treatment with acid gave a product analysing rather badly for the expected evodone. It may be a mixture of evodone and the cyclic half-acetal (XII), because the ultra-violet spectra of these two substances would not be expected to differ greatly.

Experimental

(*a*) 2:5 - *Dihydro* - 1:3 -*dimethoxybenzene.*—Sodium (12 g.) in small pieces was added gradually to 1:3-dimethoxybenzene (15 g.) in liquid ammonia (400 ml.) and ethanol (40 ml.). When the blue colour disappeared, water (200 ml.) was added and the aqueous solution extracted with ether (2 × 30 ml.). After evaporation of the solvent, 2:5-dihydro-1:3-dimethoxybenzene was distilled, b.p. 49°C./0.9 mm. (Found: C, 68.5; H, 8.7, $C_8H_{12}O_2$ requires: C, 68.5; H, 8.6).

(*b*) *Condensation of* 2:5 - *Dihydro* - 1:3 - *dimethoxybenzene with α-Bromoacetal.*—Potassium (5.5 g.) was added in small pieces to liquid ammonia (300 ml.) containing a trace of ferric nitrate. When the blue colour disappeared 2:5-dihydro-1:3-dimethoxybenzene (13 g.) in dry ether (15 ml.) was added and the mixture allowed to stand for ten minutes. α-Bromoacetal (6 g.) in an equal volume of dry ether was added. When the red colour was discharged the mixture was diluted with water (200 ml.) and extracted with ether. After evaporation of the solvent the residual oil was distilled. α-(1:4-dihydro-2:6-dimethoxyphenyl)-acetal had b. p. 62°C./1.0 mm. (Found: C, 65.4; H, 9.1. Calc. for C_{14} H_{24} O_4; C, 65.6; H, 9.4).

(*c*) *Cyclisation of* α-(1:4-*Dihydro*-2:6-*dimethoxyphenyl*)-*acetal.*—α - (1:4 - Dihydro - 2:6 - dimethoxyphenyl) - acetal was cyclised by warming on a water bath with hydrochloric acid (2N). The aqueous solution was extracted with ether and the extract rapidly washed three times with ice-cold sodium hydroxide (2%; 3 × 2 ml.) and then with water (2 × 2 ml.). The solvent was evaporated and the residual oil distilled b.p. 60°C./1.2 mm.

Treatment of this oil with semicarbazide hydrochloride gave a *semicarbazone*, m.p. 195°-6°/C. from absolute ethanol. (Found: C, 55.5; H, 6.3. Calc. for $C_9H_{11}O_2N3$, C, 56.0; H, 5.7).

The oil was regenerated from its semicarbazone and treated with 2:4 - dinitrophenylhydrazine hydrochloride. A dark red derivative, m.p. 247-8°C., from ethyl acetate, was obtained. (Found: C, 53.4; H, 4.2. Calc. for $C_{14}H_{11}O_5N_4$, C, 53.2; H, 3.8).

(*d*) *Condensation of* 2:5 - *Dihydro* - 1:3 - *dimethoxybenzene with* α - *Bromopropionacetal.*—2:5 Dihydro - 1:3 - dimethoxybenzene was condensed with α-bromopropionacetal by the method as described under (b). The condensation product had b.p. 67°C./1.2 mm. (Found: C, 66.2; H, 7.8. Calc. for $C_{15}H_{26}O_4$, C, 66.6; H, 7.6).

(*e*) *Cyclisation of Condensation Product, b.p.* 67°/1.2 *mm.*—Cyclisation of this condensation product was carried out by the method as described under (c). The oily product obtaied after cyclisation was distilled, b.p. 58°C./1.2 mm.

Ultra-violet Absorption Spectrum of Cyclisation Product.—The ultra-violet absorption spectrum of the oil, b. p. 58°C./1.2 m.m., was measured in ethanol.

$\lambda_{max.}$ 274-275 mμ; log. ϵ_{max}, 3.448.

(*f*) 1:4 - *Dihydro* - 3:5 - *dimethoxytoluene.*—This derivative, b.p. 54°C./1.4 mm. was obtained from 3:5-dimethoxytoluene by the method described under (a).

(*g*) *Condensation of* 1:4 - *Dihydro* - 3:5 - *dimethoxytoluene with* α-*Bromoacetal.*—Potassium (3.7 g.) in small pieces was added to liquid ammonia (300 ml.) containing a trace of ferric nitrate. 1:4 - Dihydro - 3:5 - dimethoxytoluene (10 g.) in dry ether (25 ml.) was added after the blue colour was discharged. The mixture was allowed to stand ten minutes and α-bromoacetal (25 g.) in an equal volume of dry ether added. When the red colour disappeared the mixture was diluted with water (200 ml.) and extracted with ether. Evaporation of the solvent gave α-(1:4-dihydro-2:6-dimethoxy-4-methyl-phenyl)-acetal, b.p. 152°C./1.3 mm. (Found: C, 66.4; H, 9.9. Calc. for $C_{15}H_{26}O_4$, C, 66.6; H, 9.7).

(*h*) *Cyclisation of* α-(1:4-*dihydro*-2:6-*dimethoxy*-4-*methyl-phenyl*)-*acetal.*—Cyclisation of the product, b.p. 152°C./1.3 mm. was carried out by the method as described under (c) above. The oil obtained after cyclisation distilled at 103°C./1.7 mm.

(*i*) *Condensation of* 1:4 - *Dihydro* - 3:5 - *dimethoxytoluene with* α -*Bromopropionacetal.*—1:4 - Dihydro-3:5 - dimethoxytoluene was condensed with α-bromopropionacetal in the presence of potassium

amide in liquid ammonia by the method described above. The crude condensation product distilled at b.p. 85°C./1.4 mm.

(*j*) *Cyclisation of Condensation Product, b.p.* 85°C./1.4 *mm.*—Cyclisation of the oil, b. p. 85°C./1.4 mm. was carried out by the method as described before. The product obtained was distilled, b. p. 76°C./1.5 mm.

Acknowledgements.—The author wishes to express his gratitude to Professor A. J. Birch for his kind help and advice and also to Dr. E. Ritchie and Dr. P. Elliott for many helpful suggestions. Finally he would like to thank the Colombo Plan Authorities for a fellowship.

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STANDARDIZATION OF SOME LOCAL AROMATIC HERBS. PART I

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(Received October 20, 1960)

Introduction

Standardization is of paramount importance to ensure the quality and potency of herbs and drugs. It is also necesary for the development of drug industry and maintenance of national health. Recognizing the importance of standards for indigenous drugs, analysis of some aromatic herbs was carried out which can be utilised to the best advantage by the local pharmaceutical industry. Such information will also help in bringing out a publication like Pakistan Pharmaceutical Codex on the lines of Indian or British Pharmaceutical Codex to set standards compatible with local needs.

Materials and Methods

Four drugs, 'ajowain', 'cumin', 'dill' and 'fennel' belonging to different genera of the family *Umbelliferae* were selected for the present work. Samples of crude herbs were collected both from the local drug market of Peshawar and from the Experimental Farm of the North Regional Laboratories. The percentage of active principles and other tests were calculated on the basis of the weight of air dried samples and their values represent the average of at least two determinations in each case. Each drug and the volatile oil contained therein were analysed according to the methods prescribed in the Indian Pharmaceutical Codex 1953 or the British Pharmaceutical Codex 1954.

Ajowain consists of the dried, ripe fruits of *Trachyspermum ammi* (Linn.) Sprague syn. *Carum copticum* Benth. and Hook, and ajowain oil is the volatile oil obtained by their distillation. The results obtained with both of them are tabulated in Table 1.

Cumin (Bengali: jira; Urdu: sufaid zira) is the ripe fruit of *Cuminum cyminum* Linn., and oil of cumin is the volatile oil obtained by steam distillation from the fruits of this cumin. The results obtained are tabulated in Table 2.

Dill (Indian) or soya (Urdu) consists of the dried, ripe fruits of *Anethum sowa* Kurz and oil of Indian dill is the volatile oil obtained by distillation from its fruits. The results obtained with this drug and its oil are given in Table 3.

Fennel or sounf (Urdu) is specified to be the dried, ripe fruit of the cultivated plants of *Foeniculum vulgare* Mill, and oil of fennel is the volatile oil obtained by steam distillation from it. The

Table 1.—Results obtained with Ajowain and its Oil.

Tests	Cultivated sample	Market sample	I.P.C. 1953 standards
Drug			
Foreign organic matter	1.7%	1.9%	Not more than 2 %
Oil			
Yield of volatile oil	0.5%	6.35%	4-6%
Specific gravity at 15°C.	0.911	0.927	0.910 - 0.930
Optical rotation	+0.9°	+0.7°	0° to +5°
Ref. index at 20°	1.497	1.499	1.498 - 1.504
Solubility (90% alcohol)	1 in 1.5 vols.	1 in 1.5 vols.	Soluble in 4 vols. of alcohol.
Thymol content (sulphite method)	45%	50%	Not less than 40%

Table 2.—Results with Cumin and its Volatile Oil.

Tests	Cultivated sample	I. P. C. 1953 standards
Drug		
Foreign organic matter	0.9%	Not more than 2%
Ash	6.2%	Not more than 8%
Voltile oil	3.5%	Not less than 2.5%
Oil		
Specific gravity at 15°C.	0.9290	0.8945-0.9300
Optical rotation	+ 4.6°	+ 3.6° to + 8°
Refractive index at 20°	1.501	1.491 - 1.507
Aldehydes	20.0%	Not less than 16%
Solubility (80% alcohol)	1 in 8 vols.	1 in 11 vols. of alcohol

TABLE 3.—RESULTS WITH DILL (INDIAN) AND ITS OIL.

Tests	Cultivated sample	Market sample	I.P.C. 1953 standards
Drug			
Foreign organic matter	0.5%	0.7%	Not more than 2%
Volatile oil	2.3%	4.3%	Not less than 2%
Oil			
Specific gravity at 15°C	0.989	0.975	0.9448 - 0.9896
Optical rotation	+48.1°	+48.1°	+41° to 48°
Refractive index at 20°	1.499	1.497	1.491 - 1.499
Solubility (90% alcohol)	1 in 0.4 vol.	1 in 0.5 vol.	Soluble in equal volume of alcohol.
Carvone content (sulphite method)	21.0%	20.7%	19.0 - 22.0%

TABLE 4.—RESULTS OBTAINED WITH FENNEL AND ITS OIL.

Tests	Cultivated sample	Market sample	I.P.C. 1953 standards
Drug			
Foreign organic matter	0.55%	1.3%	2.9%
Yield	2.03-2.17%	1.5-1.02%	Not more than 4%
Acid insoluble ash	0.82%	0.81%	Not more than 1.5%
Cold-water extractive	27.5%	25.0%	Not less than 20%
Ash	9.1%	9.7%	Not more than 12%
Oil			
Sp. gr. at 15°	0.973	0.972	0.956-0.976
Optical rotation	+13°.9	+8°.6	+ 11° to 24°
Refractive index at 20°	1.544	1.548	1.528-1.538
Solubility (80% alcohol)	1 in 7 vols.	1 in 8 vols.	Soluble in 8 vols.
90% alcohol	1 in 0.5 vols.	1 in 0.5 vols.	Soluble in 0.5 vols.
Congealing point	11.6°C.	14.6°C.	Not below 3°C.
Reaction: 1 in 8 solution of recently distilled oil in 80% alcohol is neutral to litmus paper. (I.P.C., 1953)	Positive	Positive	
Heavy metals: The oil should comply with the test for heavy metals in volatile oils. (I.P.C., 1953)	,,	,,	

results obtained with them are given in Table 4.

Discussion

A perusal of Table 1 shows that local ajowain and its volatile oil fulfill the standards of the I.P.C. 1953 and they can be used where they are specified by that codex. From Table 3 it is also evident that the local Indian dill as the drug and as oil is also up to the standard and can be used where such pharmaceuticals are required. It can also be used as a substitute for dill according to B.P.C. 1954.

As regards fennel, it is clear that it also complies with the Indian Pharmaceutical Codex requirements. Moreover it is included in British Pharmacopoeia 1953 and B.P.C. 1954, and comes up to their standards. So its use according to these pharmacopoeias is recommended in medicine and it can be utilised as an official drug. As is evident from Table 2, *Cuminum cyminum* is also a standard drug and may be used where this is prescribed by that Codex.

All these four drugs are also used in the Eastern system of medicine by Hakims and Vaids. Therefore on the basis of present investigations, the prescription of these herbs in the doses indicated in the said pharmacopoeias should produce the desired results.

Acknowledgement.—The authors are highly thankful to Col. M. K. Afridi, for his keen interest in the present work. Their thanks are also due to Mr. Abdul Hamid for his assistance in the course of this work and to Mr. S. M. A. Kazmi for supplying samples of cultivated herbs.

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Special Article

SUMMARIZED REPORT ON THE PROCEEDINGS OF THE 4TH CONGRESS OF THE PAN INDIAN OCEAN SCIENCE ASSOCIATION AND THE RECOMMENDATIONS OF ITS COUNCIL

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(Received January 15, 1961)

1. Historical Background of P.I.O.S.A.

The Pan Indian Ocean Science Association came into existence in January, 1951, and its first Congress was held the same year in India. The Second Congress was held in Australia in 1954 and the third in Madagascar in 1957. With the approval of the Government of Pakistan, the Association was invited to hold its Fourth Congress at Karachi in November, 1960. The member countries of the Association are Australia, Antarctica (France), Burma, Ceylon, France, India, Madagascar, Malaya, Mauritius, the Netherlands, Pakistan, Portugal, Singapore, Indonesia and Thailand.

The objects of the Association are:–

(i) to discuss scientific problems specially relating to the well-being of the people and the progress of the countries encircling the Indian Ocean and make recommendations to the countries concerned as and when necessary, and

(ii) to promote feelings of brotherhood among the scientists of the countries around the Indian Ocean and thus help build up closer and stronger ties of friendship among these countries.

The following broad divisions of science were included under the organization of the P. I. O.S.A.:—Section A- Physical Science; Section B– Biological Science; Section C- Geological Science; Section D - Agricultural Science ; Section E - Economics, Education and Social Science; Section F - Geography, Oceanography and Geophysics; Section G - Human Ecology. In accordance with an earlier decision of the P. I. O. S. A. Congress, seven National Committees, one for each of the above sections, were set up.

2. Inauguration

The inaugural address was delivered by Mr. A. K. Khan, Minister for Industries, Government of Pakistan. This was followed by the installation of the new General President of the Congress, Dr. Salimuzzaman Siddiqui, by the retiring General President, Prof. J. Millot of Madagasdar.

Making a reference in his inaugural address to the achievements of science, which is now reaching out to the exploration of space, the Minister for Industries emphasized that the well-being of our people still depends primarily on "our labours on this familiar old earth to win from it food for their sustenance and mineral products for their material advancement." Mr. Abul Kasem Khan also pointed out that the economically developed countries should share with Pakistan and her neighbours their greater knowledge and technical experience, in order to promote the various sectors of their national development. He said that since the advent of the new regime, everything possible was being done in Pakistan to redress the defects of the previous situation and to harness all available means for stepping up agricultural production and industrial development with a view to ensuring a balanced and well integrated economy.

In his presidential address, Dr. Salimuzzaman Siddiqui, who headed Pakistan's 10-man delegation, said that while international bodies were striving to strengthen the economies of the developing countries, regional associations such as the P.I.O.S.A. could promote a balanced progress in the various sectors of their national life due to their keener appreciation of their mutual problems. The Secretary of the P.I.O.S.A., Dr. Qudrat-i- Khuda, dealt with the problems Pakistan had been facing since her birth in 1947. He also referred to the two cyclones in East Pakistan, and added that these cyclones were a challange to science. He held the view that scientists must do something of a permanent nature to overcome such natural calamities. The retiring President, Prof. J. Millot, thanked the Government of Pakistan for their hospitality and the Minister for Industries, Mr. A. K. Khan, for inaugurating the conference.

3. Delegates to the 4th Congress

The following delegates and observers from member countries and international scientific

societies attended the 4th Congress held in Karachi from 16th to 24th November, 1960.

I. OFFICIAL DELEGATES FROM MEMBER COUNTRIES

Australia

1. Professor A. D. Ross, Leader of the delegation and founder of P.I.O.S.A., President, Australian Academy of Science.

2. Professor L. J. H. Teakle, Deputy Leader of Australian delegation, Professor of Agriculture, University of Queensland.

3. Dr. E. P. Hodgkin, Reader in Entomology and Comparative Anatomy, University of Western Australia.

4. Mr. J. J. Basinski, Research Officer, Division of Land Research and Regional Survey, Commonwealth Scientific and Industrial Research Organisation.

Ceylon

5. Dr. N. G. Baptist, Leader of the delegation, Department of Biochemistry, Faculty of Medicine, Colombo.

France

6. Professor J. Millot, Director, Institute for Scientific Research for Madagascar.

French Antarctica

7. Mademoiselle Genevieve Pillet, Bureau Scientifique des Tarres Australes et Antarctiques Francaises.

India

8. Professor M. S. Thacker, Leader of the delegation, Secretary to the Government of India, Ministry of Scientific Research and Cultural Affairs, and Director-General of C.S.I.R., New Delhi.

9. Dr. K. S. Krishnan, Director, National Physical Laboratory, New Delhi.

10. Dr. K. Chandrasekharan, Professor of Mathematics, Tata Institute of Fundamental Research, Bombay.

11. Dr. T. S. Sadasivan, Director, University Botany Laboratory, Madras.

12. Dr. L. N. Johri, Reader, Department of Zoology, Delhi University.

Indonesia

13. Professor Dr. Ir. Tojib Hadiwidjaja, Leader of the delegation, Dean, Faculty of Agriculture, University of Indonesia, Bogor, Indonesia.

14. Dr. R. P. Oetojo, Director, Central Veterinary Institute, Bogor.

15. Mr. Hasanuddin Saanin, Head of the Institute of Inland Fishery, Bogor.

16. Dr. Dradjat, Deputy Head of Institute of Nutrition, Department of Health, Djakarta.

Madagascar

17. Dr. R. Paulian, Leader of the delegation, Deputy Director, Institute for Scientific Research for Madagascar.

Malaya (Kuala Lampur)

18. Professor J. R. Hendrickson, Professor of Zoology, University of Malaya, Kuala Lampur.

The Netherlands

19. Dr. G. H. Vander Kolff, Leader of the delegation, Professor of Eastern Economics, University of Amsterdam.

20. Dr. J. Van Duijnen Montijn, Director of Oceanography and Maritime Meteorology Department, Royal Netherlands Meteorological Institute, D. Bilt.

21. Dr. J. De Wild, Professor of Agricultural Zoology (Entomology), Agricultural Institute, Wageningen.

22. Dr. H. Vlugter, Irrigation Expert to F. A. O., Punjab Club, the Mall, Lahore.

23. Dr. J. G. Vermaat, Pedologist to the F. A. O., c/o UNTAB, Sind Club, Karachi.

Pakistan

24. Dr. Salimuzzaman Siddiqui, Leader of the delegation, Director, Pakistan Council of Scientific and Industrial Research, Karachi.

25. Dr. M. Qudrat-i-Khuda, Director, East Regional Laboratories, Pakistan Council of Scientific and Industrial Research, Tejgaon, Dacca.

26. Dr. Nazir Ahmad, Textile Adviser to the Government of Pakistan, Karachi.

27. Dr. N. A. Qazilbash, Professor of Botany, University of Peshawar, Peshawar.

28. Mr. J. M. Master, Director, Geological Survey of Pakistan, Quetta.

29. Dr. S. D. Chowdhury, Director of Agriculture, Government of East Pakistan, Dacca.

30. Dr. Mahmood Hussain, Dean Faculty of Arts, Karachi University, Karachi.

31. Mr. S. N. Naqvi, Director, Meteorological Services, Karachi.

32. Col. M. K. Affridi, Vice-Chancellor, Peshawar University, Peshawar.

33. Dr. M. Raziuddin Siddiqui, Vice-Chancellor, Sind University, Hyderabad.

Portugal

34. Prof. J. Pinto Lopes, Director of Institute of Scientific Investigations.

Thailand

35. Vice-Admiral Charoon V. Bunnag, Leader of the delegation, Director of Meteorological Department, Bangkok.

36. Captain Amporn Penyapol, Royal Thai Navy, Chief of Oceanography Division, Hydrographic Department, Bangkok, Thailand.

II. OBSERVER DELEGATES

Indian Ocean International Historical Association

37. M. Andre Scherer, Director des Services, Secretaire General de l' Association Historique International de Indian Ocean. (He also represented the Reunion Island).

Naga Expedition

38. Mr. E. C. La Fond, Ministry of Food and Agriculture, Bangkok, Thailand.

New Zealand

39. Professor A. D. Ross, Leader of Australian delegation to act as an observer of the Government of New Zealand.

Royal Society of Edinburgh (U. K.)

40. Professor Emeritus, Balnagown, Golf Links Road, Albany, Western Australia.

Science Council of Africa South of Sahara

41. Professor J. Millot, to act as Official Observer.

Turkey

42. Mr. Omer M. Baykan, Assistant Professor of the Chair of Soil Studies, Faculty of Agriculture, Ataturk University, Turkey.

43. Mr. Ferit Ucarci, Assistant Professor of Geo-technic, Faculty of Agriculture, Ataturk University, Turkey.

World Meteorological Organisation

44. Mr. Muhammed Samiullah, Deputy Director, Pakistan Meteorological Services.

45. Mr. Muhammed Khalil Khan, Dupty Director, Pakistan Meteorological Services.

U. S. A.

46. Mr. David C. Rife, U. S. A.

47. Mr. Robert G. Snider, Co-ordinator, International Indian Ocean Expedition.

F. A. O.

48. Mr. Ijaz Ahmad, F. A. O. Country Representative, Karachi.

Besides the above, about 100 delegates from the universities and other scientific institutions in the country attended the Congress. Invitations were extended to well known industrialists, scientists and members of diplomatic corps, specially those representing the member countries of the P. I. O. S. A., to attend the inaugural session on the 16th November 1960.

A Reception Committee consisting of eminent scientists and industrialists was formed under the Chairmanship of Mr. B. A. Hashmi, Vice-Chancellor, Karachi University, and a number of sub-committees were also constituted to ensure effective co-ordination in the management of the affairs of the Congress.

4. Sectional Meetings

Almost all the countries surrounding the Indian Ocean are now running on a deficit with regard to the production of major food crops, and are also economically undeveloped because of lack of technical data necessary for the adequate utilization of their natural resources. It was accordingly agreed by all the scientists whithin the country and also by the members of most participating countries that the main focus of discussions in the Congress should be on food and the utilization of indigenous resources for national development. The first day was devoted to the reading of presidential addresses in the various sections, which served to provide the keynote for the discussions and for the papers that were read during the whole period of the Congress.

A. Physical Science

(*i*) *Physics*.—In the meeting of the Physical Science Section presided over by Prof. K. S. Krishnan of India, Dr. M. M. Qurashi of the Central Laboratories, P. C. S. I. R., in the course of discussions on the rapid estimation of the oil content of a single cottonseed, showed how the water content of the seed would influence the electrical properties of cottonseed and the cake. Dr. Qurashi also described the results of measurements on the intermolecular energy of activation in some hydroxylic liquids.

While discussing the effect of surface smoothness of some crystals on the thermal conductivity measurements, Dr. Tirmizi of Karachi University showed that these measurements were sensitive to the nature of surface of the crystals. Dr. Mujtaba Karim of Karachi University presented two interesting papers on the determination of atmospheric radio-activity and its correlation with weather conditions, nuclear explosions and other phenomenea, for example the solar eclipse.

The Chairman, Dr. K. S. Krishnan, gave a brief review of some recent researches on the fundamental side being carried out in his laboratories, with special reference to an elegant new method for determining the absolute value of the thermionic emission co-efficient for various substances.

Mr. S. N. Naqvi of the Meteorological Department, presented a series of papers, one of which dealt with periodicity in the cyclonic storms and depressions which cause floods in the Indus basin.

(*ii*) *Chemistry*.—The majority of the chemical papers were of an applied character, dealing with the better utilization of indigenous raw materials and industrial waste products. Particular mention must be made of papers on (1) preparation and application of betulin-based modified rosin esters by Mr. M. Aslam; (2) Copper fungicide based on rosin by Mr. M. Aslam; (3) Synthetic tanning material from black (lignin) liquor by Dr. E. Dubash; (4) Examination of indigenous bentonites for refining cottonseed oils by Dr. M. K. Bhatty; (5) Survey of indigenous aromatic plants by Mr. Shafiq.

B. Biological Science

In the Biological Section's meetings, which were presided over by Dr. Millot of Madagascar, sea fish and inland fish resources in Pakistan and their development were exhaustively discussed. Dr. Millot presented a number of papers on zoological and botanical work carried out in the Herquelen Island in the south of Indian Ocean. Mr. S. Saanin, an Indonesian delegate, read papers describing results obtained regarding the effects of fertilization on the luminology of paddy fields and of the toxicity of insecticides on the cultivation of fish in Indonesia.

C. & F. Combined. Geology, Geography, Oceanography and Geophysics

Climate.—In the combined sectional meetings of geology, geography, geophysics and oceanography, presided over by Vice-Admiral C. V. Bunnag of Thailand, Dr. Mentia described the need of international cooperation for compilation and publication of information on climatology and the climatological Atlas of the Indian Ocean. Prof. M. I. Chowdhry of Pakistan described his findings based on extensive studies of morphological changes in the Bengal Basin. Dr. David D. Smith described the work on the geomorphic studies of the Makran coast and Lasbella area in West Pakistan. He also mentioned investigations on beach rocks which may be described as cemented beach sand of very recent origin.

Mr. M. M. Rizki of the the Meteorological Department read a paper on the chlorinity and salinity of the Arabian Sea and Bay of Bengal, based on analysis of the samples collected by P. N. S. Zulfikar. Mr. A. H. Qureshi of the Survey of Pakistan gave a history of survey and cartography in Pakistan and of the important programmes the Survey of Pakistan is engaged in.

D. Agricultural Science

Under the presidentiship of Prof. L. J. H. Teakle and Dr. S. D. Chowdhury, the Agricul-

tural Section discussed papers on food and food Products. Dr. C. M. Ishaq of the North Regional Laboratories, P. C. S. I. R. described the fruit resources of Pakistan and emphasised the enormous possibilities of building up the economy through proper processing of the fruits. Dr. Bhatti in a paper on commercial vinegar manufacture demonstrated that with the raw materials available in the country there exist good possibilities of developing this industry on an economic basis.

Dr. H. N. De of the East Regional Laboratories, P.C.S.I.R., discussed the starch resources of East Pakistan, their potential amylose and amylopectin contents and other properties which would suggest the suitability of these starches for industrial and edible uses. In presenting the papers on the biogenesis of starch and vitamin C in two varieties of potato, Dr. De also showed that, although the white skin variety possessed more starch, it contained less vitamin C than the red skinned one. In the discussion on the effect of utensils used for the cooking of vegetables, it was pointed out that all types of utensils, earthen, aluminum and iron, cause a loss of about 65 per cent of vitamin C.

Dr. D. K. Das presented a paper on the mangrove plants of East Pakistan and emphasised the importance of vegetation in conservation of the coastal area by preventing erosion, and the use of such plants as raw material for newsprint manufacture and also for fuel.

In dealing with the longer storage life of parboiled rice it was shown by Dr. De of the East Regional Laboratories that this is mainly due to inhibition of any loose acitivity in the parboiled rice for which less reducing sugar is formed leading to a retarded growth of the microorganisms. It was further shown that parboiled rice requires smaller quantities of water for cooking and also the gruel water left from it was lower in starch content as compared to the cooking of raw rice.

In discussing the nutritive value of sundried 'Sukti' fish, it has been reported that the application of different spoilage tests, as developed in the East Regional Laboratories, shows no evidence of spoilage in this traditionally processed foodstuff of East Pakistan.

A report about the high protein formulated diets prepared for improving the nutrition of school-going children was submitted by Dr. De. He mentioned that fish flour, egg powder and the vermicelli prepared by blending these protein sources with the sweet potato starch by a new technique can withstand severe climatic conditions without showing spoilage.

E. Economics, Education and Social Science

In the section of economics, education and social sciences, which met under the presidentship of Dr. G. H. Vender Kloff of Amsterdam, two significant papers relating to (1) fear arousing communication, and (2) juvenile delinquency, were read and discussed.

G. Human Ecology

(*i*) *Nutrition.*—Papers read at meetings of the Human Ecology Section were mainly devoted to the nutritional problems in the countries surrounding the Indian Ocean. Dr. N. G. Baptist of Ceylon, who presided over the meeting of this section, discussed the protein and amino acid requirements of the people of this region. Dr. Dradjat of Indonesia showed how the nutritive value of food may be improved by fermentation processing.

Dr. A. K. M. Wahed in his paper gave an account of the problems of old age in their social contact and suggested that home for the care of old and the aged should be instituted.

Problems of man's existence in space were mentioned by Dr. Musa Khan and he described how air could be fairly well supplied by algal cultivation in the space ship.

Dr. Ilyas brought to the notice of the members the alarming situation about the malnutrition of school children as revealed in the surveys.

(*ii*) *Irrigation Problems.*—In the joint session of agriculture and human ecology, held under the presidentship of Prof. L. J. H. Teakle, Dr. Vlugter discussed the irrigation problems for agriculture in West Pakistan, and pointed out the need for utilising the available water in smaller areas but with fuller benefit for irrigation and drainage rather than using the water to only inadequately serve larger areas, which will then suffer more and more from salinity problems. He also pointed out that the water available in the rivers should be used for irrigation and drainage only to the extent that it can efficiently carry the sediments to the sea. If no consideration to this is given, the floods would be constantly on the increase. He also mentioned that the tube-well project of irrigation will help solve the salinity problem to a certain extent.

5. Council Meetings and Recommendations

At the meetings of the Council of P. I. O. S. A., consisting of heads of delegations from the member

countries, held on the 16th and 22nd November, 1960, a number of recommendations were discussed, and were taken up for consideration at the final session of the Congress on the 24th November, 1960, at which the following resolutions were unanimously adopted.

Resolutions

(*a*) *PIOSA Secretariat:*

1. The Congress, feeling the need for a Secretariat, decided: (a) to form a Secretariat, expenses of which will be met by voluntary contributions from the member countries; (b) to locate this secretariat immediately at Karachi in Pakistan.

2. It also decided that a Working Committee of four, consisting of (i) Dr. S. Siddiqui, (ii) Prof. A.D. Ross, (iii) Prof. M. S. Thacker and (iv) Prof. J. Millot, be entrusted with the drafting of suitable rules for the working of PIOSA and its Secretariat. The draft rules should be circulated to the members of the Council before the end of May, 1961, and the draft be finalized before May, 1962. The draft rules will be placed before the fifth Congress for ratification.

3. Pending the ratification of these rules, the office of the Secretariat will be governed by such of them as will be applicable to it.

4. The Secretariat will work under the guidance of the President, and will be responsible to the Council.

5. Resolved that the Secretariat of PIOSA collect and distribute among member countries information relating to scientific matters of topical interest, in cooperation with other appropriate bodies, and that, to this end, it bring out a quarterly bulletin or a news letter.

6. Resolved that the Secretariat work out some modus operandi to facilitate the obtaining of scientific materials, microfilms, exchange of publications, reports on the educational systems in these areas, preparation of a Bibliography of the Indian Ocean areas, etc., and to facilitate the expeditious supply of such materials to the member countries.

(*b*) *Other matters:*

7. The Congress noted that, after the adoption of resolutions 10, 11 and 12 by the third Congress held at Tananarive in 1957, extensive oceanographic studies have been projected by the International Council of Scientific Unions (ICSU) through its special Commission, SCOR, and that high priority has been assigned to the study of the Indian Ocean. Resolved that member countries desirous of participating in the ICSU project may get in touch with ICSU and SCOR through appropriate national agencies, and that PIOSA lend whatever support it can to the Indian Ocean Expedition; also that the Secretariat be asked to collect all relevant information in this connection and make it available to the member countries.

8. Resolved that the Working Committee take such measures as may be necessary for effecting liaison with the Pan Pacific Ocean Association, the Scientific Council for Africa and similar other organizations, and arrange for exchange of information with these bodies.

9. Resolved that the Working Committee explore the possibilities as to the venue of the Fifth Congress, and of the following two Congresses if possible, make a recommendation at an early date, but not latter than July, 1961, and submit it to the Council for decision.

10. Resolved that the scientific programme of future Congresses should comprise symposia, invited addresses and research papers, the organisation of which programme should be entrusted to a Programme Committee, to be appointed by the Working Committee of the Council after the venue of the Congress has been decided upon. Further resolved that the work of the Congress be organized under three major heads, namely:

(i) Physical Sciences,

(ii) Biological Sciences (both basic and applied),

(iii) Social Sciences,

which between them will cover all the subjects coming under the purview of the present seven sections.

11. The Congress resolved that a panel of experts in the different scientific fields covered by the work of PIOSA be constituted, whose advice may be sought by the Secretariat and the various committees of PIOSA.

12. The Congress noted that in the teaching of science at the secondary school level, the biological sciences do not seem to receive at present the same attention as the mathematical and physical sciences, and wishes to emphasize the importance of keeping a suitable balance between the mathematical, the physical and the biological sciences.

The Congress considered a letter from the Secretary General of International Union of Geodesy and Geophysics, inviting PIOSA to nominate a scientist for liaison work with International Union of Geodesy and Geophysics. The Congress resolved that Mademoiselle G. Pillet be nominated as the liaison scientist, it being under stood that this appointment will not involve any financial commitment for PIOSA.

6. Visits and Tours

While arrangements were made for the delegates to visit scientific places of interest, *i.e.*, Basic Medical Sciences Institute, the Laboratories of the P. C. S. I. R., the Fish Harbour, the Karachi University and the Cotton Research Institute, they were also taken on recreation trips to Sandspit on the 18th November, 1960.

The P. C. S. I. R. extended full hospitality during the Congress to the leaders of the delegations and provided lodging to official delegates from the member countries participating in the Congress. Adequate arrangements were also made for providing transport facilities to the delegates, and the Ministry of Finance is to be thanked for allowing an estimated expenditure of Rs. 50,000, which was to be met out of the overall budget allocation of the P.C.S.I.R. The delegates were invited to a dinner by the Atomic Energy Commission, to tea by the Karachi University, also by the Association of Scientists and Scientific Professions, and to a reception and a farewell dinner by the P.C.S.I.R.

SHORT COMMUNICATIONS

OBSERVATIONS ON THE VOLTAGE DEPENDENCE OF THE INSULATION RESISTANCE OF SOME INSULATING MATERIALS

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(Received December 2, 1960)

In the course of testing the electrical insulating properties of several materials prepared in the Central Laboratories, from indigenous ingredients, it was considered of interest to measure their insulation resistance at various voltages below the breakdown limit. The tests were carried out with A. C. voltages and the apparatus used for these measurements was put together from components obtained locally. The set up is shown diagrammatically in Fig. 1. The special features are the limiting resistor R_1 in the primary circuit of the high-tension transformer and the current measuring resistor R_2 in parallel with the voltmeter V on the earthed side of the sample under test in the high-tension secondary circuit. The resistor R_1 serves to limit the primary current to one Ampere so as to prevent overloading of the high tension transformer when the sample under test breaks down, while R_2 (about 20 kilo-Ohms) does double duty as a current measuring resistor (before breakdown), and as a safety resistor ensuring continuous permanent contact on the earth side (e.g. when changing ranges on the meter in parallel with R2). The current 'I' is calculated by the simple formula

$$I = V\left(\frac{1}{R_2}+\frac{1}{R_V}\right)$$

$$= \frac{V}{R_2}\left(1 + \frac{R_2}{R_V}\right)$$

where R_V is the internal resistance of the voltmeter, and $(R_2/R_V) << 1$, except when the voltmeter is on the lowest 10 volt range. The r. m.s. kilovolts were read directly on the 'kV' meter the series resistances of which are connected across the high-tension secondary; a small correction for the voltage drop 'V' was made in the few cases just before breakdown where it was significant. The test electrodes were of the standard type described in B. S. S. No. 634: 1935, the lower one being a flat brass disc 3 inches diameter and 1 inch thick, and the upper one a solid brass cylinder 1½ inches diameter and 1½ inches high. The various sharp edges were rounded to the recommended radius.

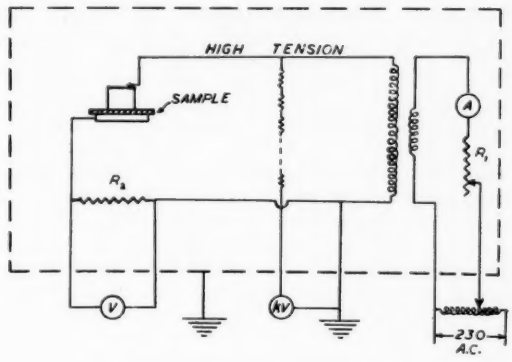

Fig. 1.—Diagrammatic sketch of the apparatus used for measuring the resistance of insulating materials upto their breakdown limit.

A set of measurements taken with a 2.45 mm. thick sample of "Jutoid", a rubberoid type of material based on jute and developed in the C. S. I. R. laboratories, are given in Table 1, while the graphs of Fig. 2(a) and (b) show plots of insulation resistance against kilovoltage obtained with two differently prepared samples of this type of material. The points at the very lowest applied tension of one kilovolt are subject to a rather large experimental error because of the very small currents, and the range of this error is indicated by the short vertical lines in Fig. 2.

The graphs of Figs. 2(a) and (b) both show the interesting feature that, after the initial rapid drop in resistance, there is an extended region in which the resistance varies linearly with applied voltage. What is more, this straight line when extrapolated towards zero resistance yields a voltage that is of the same order as the breakdown voltage. The ratio of the Breakdown Voltage to this extrapolated voltage is 0.88 ± 0.05 for the particular samples tested in the two graphs of Fig. 2. Further evidence for this linear variation of resistance is provided by the graphs of Fig. 3, which show the experimental results obtained with two different compositions for insulating varnish, applied on a standard paper to give a total thick-

TABLE I.—MEASURED INSULATION RESISTANCE FOR A 2.4 MM. SAMPLE OF "JUTOID" WHICH BROKE DOWN AT 5.1 KILO VOLTS.

Applied Voltage (kV)	1.0	2.0	2.5	3.0	3.5	4.0	4.5	4.75	5.0
Current = I (in milliamps)	0.031	0.10$_4$	0.15$_4$	0.23$_1$	0.31$_9$	0.42$_8$	0.66	0.86	1.13
Resistance = (kV/I) Meg Ohms	32	19	16.2	13.0	11.0	9.3	6.6	5.6	4.9
Specific Resistance × 10-9 (Ohm cm.)	1.6	0.88	0.75	0.60	0.51	0.43	0.31	0.26	0.23

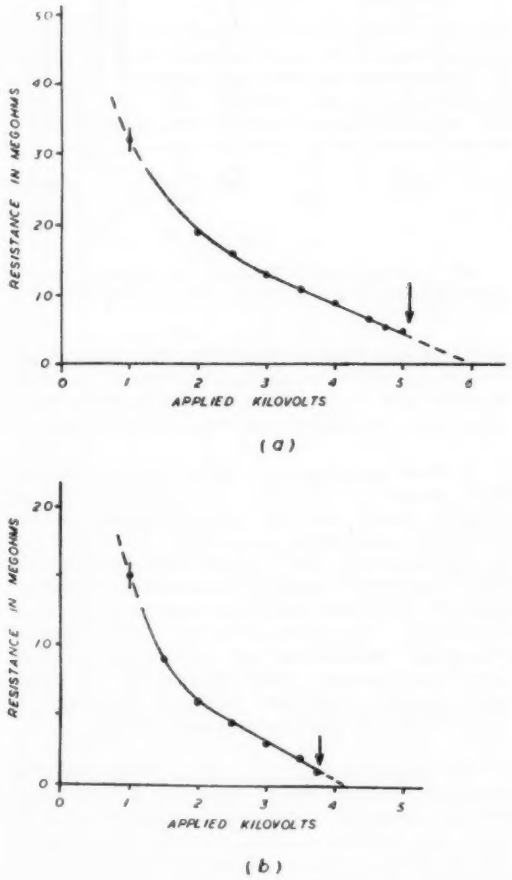

Fig. 2.—Graphs showing the variation of insulation resistance against applied kilovolts for two differently prepared samples of Jutoid, about 2.4 mm. thick. The vertical arrows indicate the breakdown voltages.

ness of very nearly 5 mills. In this case, the breakdown voltage is nearly 0.76 ± 0.05 times the linearly extrapolated value. Thus, on the basis of Figs. 2 and 3, a mean value for this factor would be 0.82 ± 0.06.

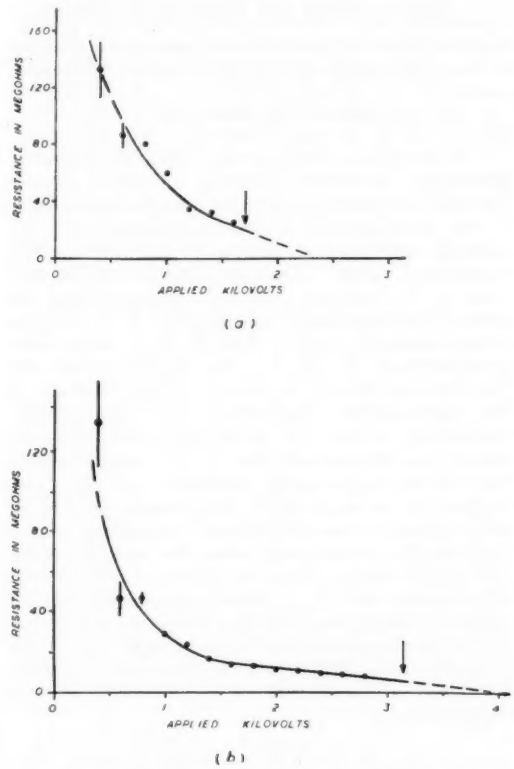

Fig. 3.—Plots of resistance against applied kilovolts for two samples of insulating varnish applied on standard thickness of paper. The vertical arrows indicate the breakdown voltages.

These findings indicate the possiblity of using this type of extrapolation as an easy means of non-destructively obtaining a measure of breakdown voltage, *i.e.*, electrical insulation strength. Further tests on other materials are in hand with a view to generalizing this and studying the precise laws of the variation of insulation resistance for a variety of materials.

The samples used in the above tests were kindly provided by Mr. M. Aslam of the Paints and Plastics Division of the Central Laboratories.

A NOTE ON THE OCCURRENCE OF PULLET DISEASE: AVIAN MONOCYTOSIS IN EAST PAKISTAN

M. WAZED ALI
Poultry Disease Investigation Laboratory, Dacca

(Received April 27, 1959)

Jungherr (1952), citing a poultry disease investigation work of Beaudette, described a condition that affected highly productive adult fowls. Sudden death with cyanosis of the comb and wattles and comparatively low flock mortality were the spectacular clinical features. On post-mortem examination the internal organs presented congestion with echymotic haemorrhage on the heart and abdominal fat with evidence of necrosis in the liver. Similar conditions were studied in various breeds of poultry by Jungherr and Levine (1940), Hurt (1941) and Gordon & Blaxland (1945), who also observed the lesions mostly in laying pullets.

Occurrence of the Disease on the Farm.—The first carcase of a cross-bred pullet, autopsied on August 21, 1958 at the Sitakundo poultry multiplication centre, presented fairly good physical conditions with apparent dehydration of muscular tissue and perceptible damage to the liver and kidney.

Again on October 18, 1958, another carcase of Jungli pullet of about 6 months of age, was sent in this laboratory for inquiry into its cause of death. The bird was fed with whole paddy, broken wheat, kheshari dal, meat offel, oilcake, local vegetables, etc., with usual drink and pasture. The post-mortem examination revealed marked darkening of the comb and wattle with fair general condition. The skeletal muscle showed extreme dehydration and patches of degeneration with crop & gizzard distended with sour smelling food material.

The proventriculas and intestine were flabby and showed catarrhal enterities with viscous mucoid contents in the lumen of the gut. No parasite was present. The liver was necrotic and presented multiple petechial haemorrhage. Kidney was enlarged. The physical change in the blood seemed to be increased viscosity and coagulability. The monocytes appeared larger with round nuclei, which indicated immaturity of the blood cells when examined under microscope. Usual bacteriological and transmission experiments of suspected material, carried out on suitable nutrient media and susceptible animals, failed to produce any positive result.

The available data on post-mortem, bacteriological and pathogenesity tests clearly ruled out the possibilities of any transmissible oetiological agent bacterial or otherwise, which supports the findings of Jungherr (1952) and other earlier workers in Avian monocytosis. Besides, they observed the ailment in young pullets on lay. It has been indicated by Morrison (1956) and Maynard & Loosli (1956) that wheat and barley may contain enough nitrate to cause poisoning to animals fed. It may, however, be stated that nitrates might have been transformed into nitrites (NO^2) in the degestive tract. These nitrites when absorbed into the blood would change the haemoglobin into methemoglobin, which could not transport oxygen through the blood stream. Paranchymatus cells of the liver, renal tubuti and other neighbouring glandular tissues were thus affected with deposit if CO_2 for want of supply of oxygen in the system. Consequently the skeletal muscle became dehydrated and the hepatic and renal tissues nectoric. Sudden death of the bird was, therefore, caused by the detrimental effect of nitrites on the involuntary cardiac muscle.

Summary.—Pullet disease or avian monocytosis was diagnosed in East Pakistan. The ailment seems to exist mostly in imported strains of fowls and is limited to certain age group. No biological factor, bacterial or otherwise, could be assigned as the direct cause of death, but the visible evidence tend to lay blame on food supplied.

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BOOK NOTICES

Proteins. Proceedings of the IVth International Congress of Biochemistry, Vienna, Volume VIII. Edited by H. Neurath and H. Tuppy. 272 pp. Pergamon Press, London, 1959. Price 70s. ($11).

There are 39 communications, many with summaries of discussions that took place after the papers were read. A general report on the proceedings is given by Neurath at the end of the book from which the trend appears to have been "the elucidation of the relation between the biological activity and the chemical structure of protein;" nothing could have superseded it. The papers are by scientists of international reputation, a couple of them being even Nobel laureates. Szent-Gyorgyi with C. Cohen has a paper on muscle proteins which ultimately aims at the study of heart physiology. H. Theare!l, another Nobel laureate, explains "the modern trends in flavoprotein research" which elucidates the structure of many an enzyme system. It is well known by now that the so called trace elements really constitute metal protein complexes and function as mettaloenzymes. B. L. Vallee has a paper of 25 pages mentioning among others that cadmium, by now, is to be one of hem. In the discussion Edsall rightly points out that "the finding of a place for cadmium among natural protein structures helps to fill a gap in our knowledge" of the role of trace elements in animal physiology. Perhaps no study can be more fundamental than that of biogenesis of proteins. A contribution in this field appears as an introduction by F. Lippmann and a longer paper by B. A. Askonas with others entitled "protein synthesis in cell free systems." Other articles are likewise indispensible and the book is worth its money and should be available to every worker on biochemistry—S. M.

All Indian Pharmaceutical Directory. Edited by M. L. Khorana, G. B. Ramasarma, A. V. Modi and M. Dalvi, on behalf on the Indian Pha:maceutical Association. Second edition, 1960, Bombay. Text covers 458 pages and includes two charts, advertisements 48 pp. Price Rs. 38.50.

The complication is in its second edition, which automatically speaks of its having already justified itself. It is in fact an extension of the activity on the part of the compilers as editors of the Indian Journal of Pharmacy, a publication of over 10 years' standing. The numerous advertisements give useful information as to manufactures of machinery, bottles and other acessories so necessary for the pharmaceutical industry. Besides this, there is a complete list of manufactured products and of their producers.

Of great practical value is the list of Indian medicianl plants and charts showing their geographical distribution. A list of some 85 pages gives addresses of pharmaceutical m nufa turers including those of Unani and Ayurvedic drugs. A supplementary list of 155 pages gives the names of 188 firms distributed in 41 important towns of India. Another list of 13 pages gives the names of suppliers of vegetable drugs. All this goes to make th bo:k an indispensible hand-maid to the pharmaceutical industry and its rapid rise in India cre:ted a demand for such a publication.

The book as a whole is a model for a similar compilation in Pakistan, but the publication under review can even otherwise prove of great profit on account of its contents. We can heartily recommend the book to the retail as well as to the whole-sale dealers of pharmaceuticals even in Pakistan. The price is by no means high for the excellent get up with a copious index.—S.M.

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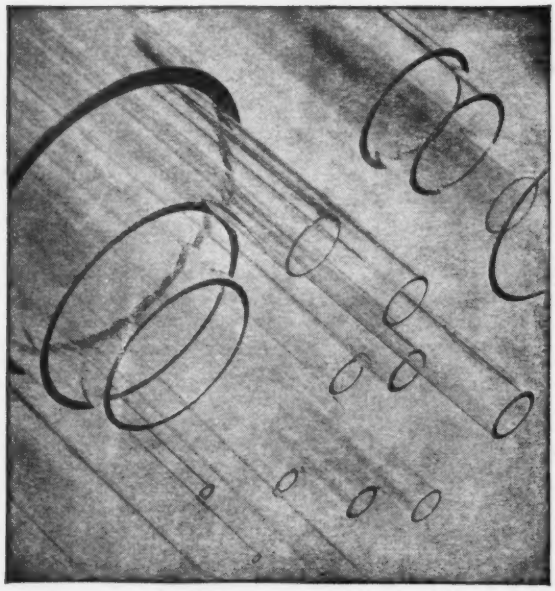

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